Probing matter under extreme conditions at the free-electron-laser facilities: the TIMEX beamline

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ABSTRACT

FERMI@Elettra is a new free-electron-laser (FEL) seeded facility, able to generate subpicosecond photon pulses of high intensity in the EUV (extreme ultraviolet) and soft x-ray range (up to 62 eV for the present FEL1 source, extended to ~ 300 eV with the FEL2 source under commissioning). Here[1] we briefly report about layout, initial results and perspectives of the TIMEX end-station, conceived in the framework of a collaboration between the ELETTRA synchrotron and the University of Camerino. The TIMEX end-station is a branch of the EIS beamline, and is specifically designed to exploit the new FEL source for experiments on condensed matter under extreme conditions. The potential for transmission, reflection, scattering, as well as pump-and-probe experiments is briefly discussed taking into account that FEL pulses can heat condensed matter up to the warm dense matter (WDM) regime. The present experimental set-up and some examples of experiments performed during the commissioning stage are presented. The dependence of the x-ray transmission and reflection as a function of the incident fluence (up to 10-20 J/cm^2) is compared with calculations. We also report about near- edge x-ray absorption data collected exploiting the full wavelength tunability of the FEL source. Perspectives for pump-probe experiments using both FEL and optical pulses, presently under development, are also mentioned.

Keywords: free electron laser, extreme conditions, warm dense matter

1. OVERVIEW

Several beamlines have been designed for exploiting the unique features of the new seeded free-electron-laser (FEL) user facility (FERMI@Elettra) available at Sincrotrone Trieste since early 2011 (see for example[1,2] and refs. therein). Three of them are currently operating and continuously upgrading their performances: coherent diffraction imaging (DIPROI), materials under extreme conditions (EIS-TIMEX), gas phase and cluster spectroscopy (LDM). The FERMI@Elettra facility includes two undulator chains (FEL1 and FEL2) covering two different spectral ranges (12.4-62 eV for FEL1, 60-310 eV for FEL2). At the present stage of development, FEL1 can operate providing nearly transform limited subpicosecond (~ 0.1 ps) pulses with a repetition rate of 10-50 Hz and energy per pulse exceeding 300 μJ. FEL2 has been already tested, is presently under development and should be operating in 2014.

The scientific program of the TIMEX end-station was conceived to exploit the FERMI@Elettra FEL source for studies of condensed matter under extreme conditions.[3–5] As an example, intense and ultrashort FEL pulses were used for creating and investigating matter in, or near to, the warm-dense-matter (WDM)[6] regime at FLASH (Hamburg).[7,8] Matter under extreme

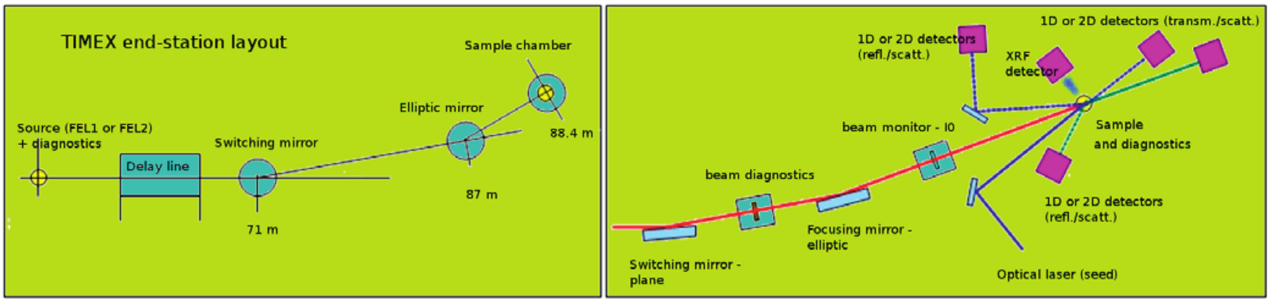

Figure 1

Sketch of the TIMEX end-station under commissioning at the FERMI@Elettra FEL facility. The left side shows the main components of the beamline, including the delay line and the elliptic mirror that should be installed after a performance test, within a few months. In the right side, we show some details of the focusing and aligning devices and the detectors used for reflection, transmission, scattering and x-ray emission/fluorescence (XRF) measurements using both FEL and optical laser pulses. The pump-probe scheme should be tested within a few months.

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conditions is also part of the scientific case of the LCLS (MEC beamline, Stanford) and XFEL (European X-ray FEL, Hamburg) facilities. In single-shot FEL experiments, a large fraction of the electrons of the specimen are excited within the pulse duration, raising the temperature of the specimen. A typical sample equilibrates its temperatures within a few picoseconds and can reach very high temperatures (up to 10^3-10^5 K) still maintaining typical densities of condensed matter (WDM regime). This state of matter is poorly known and exceedingly difficult to study, while its knowledge is of basic interest because such disordered states are those found in the interior of large planets and in stars.

The availability of a source of intense, ultrashort and monochromatic tunable pulses like FERMI@Elettra opens the way to a variety of experimental possibilities for probing condensed matter under extreme transient conditions. The use of FEL radiation is particularly promising also because it extends the ultrafast techniques already available using optical lasers to homogeneously bulk-heated specimens, opening new perspectives to study the dynamics of transitions (melting for instance) in ordered and disordered condensed matter. Moreover, ultrafast experiments give access to presently unreachable states of matter ("no man's land") because of their extremely fast transition rates. Here we report about the present status of the TIMEX end-station and some results obtained during the first days of activity, mentioning also perspectives and future plans.

2. THE TIMEX END-STATION: DESIGN AND EXPECTED PERFORMANCES

The FEL pulses produced by FEL1 or FEL2 are delivered to the beamlines through a dedicated system (PADReS[1,2,9]) for diagnostic and intensity tuning, under continuous development and upgrade. A gas attenuation chamber is available to adjust gradually the FEL pulse intensity. Two low-pressure ionization chambers placed before and after the gas chamber are calibrated to provide measurements of the intensity (I_0) at the FEL exit for each individual pulse. A 200 nm flat Al filter is available within the FEL beam transport section to eliminate the seed laser contribution.

A suitable optics has been designed for the TIMEX end-station providing unique beam-shaping capabilities for obtaining a 3-50 μm spots with the desired energy (and fluence) deposited on the sample.[5] We have also developed a novel diagnostics for the temperature reached by the sample after the pump pulse, as described in previous papers.[10,11]

As shown in Fig. 1, the beamline design is conceptually simple and includes a delay line (30 ps), a plane mirror (in future with active piezo benders), an elliptic focusing mirror with focus at 1.4 m at the sample position inside the main UHV (Ultra-High-Vacuum) TIMEX chamber. At the time of writing the delay line and the focusing mirror are still to be delivered or tested. Various devices for adjusting the intensity of the pulse and to align the beam, like insertable filters and active or passive screens, can be used along the beamline. The sample environment and main TIMEX chamber (see Fig. 1, right panel, and Fig. 2) have been kept very flexible and can accommodate various possible configurations for single-shot experiments including simple EUV and soft x-ray absorption/reflection, x-ray emission/fluorescence (XRF),[12] and pump and probe experiments[13] using either an optical laser or the FEL pulse (and its harmonics).

Figure 2

Picture of the TIMEX chamber installed and aligned at the exit of the FEL1 source in 2012. The FEL beam (blue dashed line, guide for the eye) has been aligned up to the main TIMEX chamber, where the sample position can be controlled with a 5-axis motorized manipulator while the transmitted and reflected pulses were measured by the photodiodes and thermopiles.

The TIMEX chamber installed and aligned along the FEL beam is shown in Fig. 2 (temporary installation in 2012). The experimental chamber (Fig. 2), of cylindrical shape (internal diameter: 500 mm), guarantees a vacuum level of 10^{-7} mbar. The current experimental set-up, shown in Fig. 3, consists of a 5-axis sample holder, a telemicroscope, a focusing mirror and 3 detectors (2 photodiodes, 1 thermopiles). The sample is mounted on a motorized sample manipulator stage, conceived for single-shot measurements at 10-100 Hz rate and allowing precise alignment of the sample in the interaction region with pump and probe ultrashort pulses.

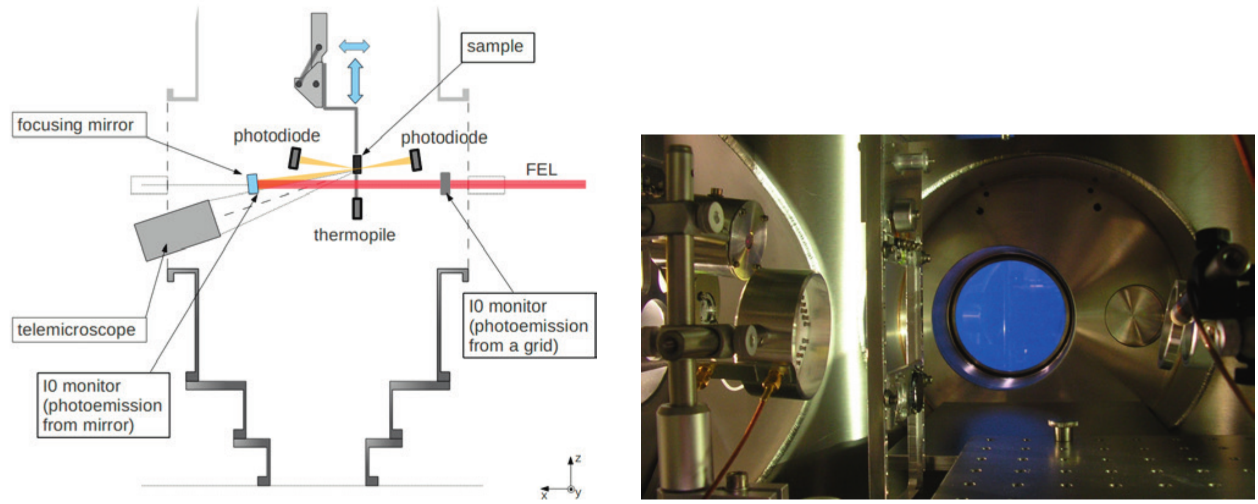

Figure 3

Left side: sketch of the current (March 2013) experimental set-up of the TIMEX chamber, including optics, detectors and diagnostics. Right side: picture of the setup including the sample holder (center), the Au grid (I_0 monitor) and a photodiode (left), and the focusing mirror (right).

The telemicroscope (resolution better than 10 μm at a distance of 35 cm) is used to determine the focal plane of the focusing mirror and to estimate in-situ the size of the FEL spot (both on PMMA specimens and fluorescence screens). The detectors allow for transmission/absorption and self-reflection intensity measurements as well as for direct measurements of the collimated primary beam intensity (Fig. 3). Additional diagnostics have been developed very recently, including a couple of devices that monitor the intensity of the incident (I_0) FEL pulses inside the chamber. Measurements of the incident I_0 in the vicinity of the sample are very important in order to eliminate possible alterations, due to the optics or to the pulse shape, with respect to the I_0 measured by the PADReS ionization chambers placed near the FEL source. Available diagnostic include also an infrared pyrometer,[10, 11] but further space is left for additional instrumentation.

The FEL beam is currently focused by a spherical mirror (Si substrate, metallic or multi-layer coating, diameter 1.5 inches, f=200 mm, angle of incidence 3 degrees). The best focus in the TIMEX chamber has a diameter of about 10 μm FWHM (80-100 μm^2) as measured on a YAG screen. However, the mirror attenuates the pulse intensity up to one order of magnitude, depending on the photon energy and coating of the mirror, due to the quasi-normal incidence of the beam. Moreover, the spherical mirror can introduce a slight stretching of the pulse (up to 0.5 ps) that can be taken under control by limiting tilt, alignment and focus of the mirror and sample positions. In the first experiments, we took particular care about geometry and alignment so that the pulse stretching introduced by the mirror was estimated to be negligible. Of course, these limitations will be overcome when the final TIMEX focusing optics (Fig. 1) will be made available.

The set-up shown in Fig. 3 has been used since the beginning of 2012 for the performance of the first preliminary experiments described in the following section, in view of the installation of the final focusing optics, delay line, and pump-probe devices.

3. FIRST EXPERIMENTS

A simple class of experiments that can be carried out using the currently available TIMEX configuration involves the measurement of the intensity of the transmitted or reflected FEL ultrashort pulses of selected specimens as a function of the incoming fluence. As mentioned in the preceding section, the FEL pulses generated by FEL1 or FEL2 sources give rise to high levels of incident fluence and deposited energy when proper focusing is achieved. The amount of deposited energy obviously depends on various factors related to the source, optics (number of photons, photon energy, spot dimensions) and target (material, thickness). Transmission and reflection measurements contain of course important information about the excited state reached by the specimens. In a previous paper[4] we reported estimates of the energy deposited in condensed matter, using pulse parameters compatible with the performances of the FEL1 source and the optics of the TIMEX end-station. Bulk heating and typical electron temperatures Te ~ 1-10 eV can be reached in ultrathin foils of selected materials. Self-standing foils of thickness in the 50-300 nm range can be produced and have the suitable robustness and reliability needed in real single-shot experiments.

In monochromatic photon transmission measurements, non-linear deviations from the Beer-Lambert law are known to occur as an effect of an increased intensity of the incident energy density of the electromagnetic field. Saturable absorption has been first observed for soft x-ray using FEL pulses by Nagler et al.[7] as shown in Fig.4 (upper-left panel). In that pioneering experiment, single-shot transmission data of a 53 nm Al foil were collected using 92 eV FEL ultra-short (15 fs) photon pulses up to fluences in the 200 J/cm^2 range. The Al L^{2,3}-edge energies are 73.1 and 72.7 eV respectively, so the kinetic energy of photoelectrons is about 20 eV. In order to observe saturation phenomena, the pulse duration must be short enough to compete with the lifetime of the excited states. For sufficiently high incident fluence, atoms in the ground state of the target become excited at such a rate that there is insufficient time for them to decay back to the ground state, and the absorption subsequently saturates (increasing the transmission). We have applied a non-linear dynamical model[14] to calculate the transmittance, obtaining a very good agreement in a wide range of fluences. We used a three-state model, constructed defining suitable ground, excited and transient states. A computer code was developed to solve the time-dependent coupled non-linear equation for the absorption process, fully related with the dynamics of the laser field. In Fig. 4 (lower-left panel) we report the preliminary results of an experiment carried out at the TIMEX end-station, using the FEL1 source tuned a photon energy of 23.7 eV (first harmonic). The effect of a repeated exposition to the FEL pulses (including also the laser seed ones) on an aluminum self-standing 100 nm foil can be appreciated in the upper-right picture of Fig. 4. The damage extends to a region with lateral dimensions of a few tens of µm. The two-dimensional map of the pulse intensity at focus (see lower-right picture in Fig. 4) shows that the lateral dimensions of the pulse at focus is about 10x10 µm (FWHM). Due to the limited efficiency of the optics, the maximal fluence reached is in the 20 J/cm^2 range. The trend obtained for the transmission curve shown in Fig. 4 and the agreement with the calculation shows that we have an indication for the presence of saturation phenomena in this range. At this energy (23.7 eV) the excitation involves valence electrons, but the kinetic energy of photoelectron is very close to the previous experiment. The trend of transmission is similar in both cases, with an almost linear increase in logarithmic scale in the low fluence side, and an asymptotic transmission at high fluence depending on different absorption phenomena.

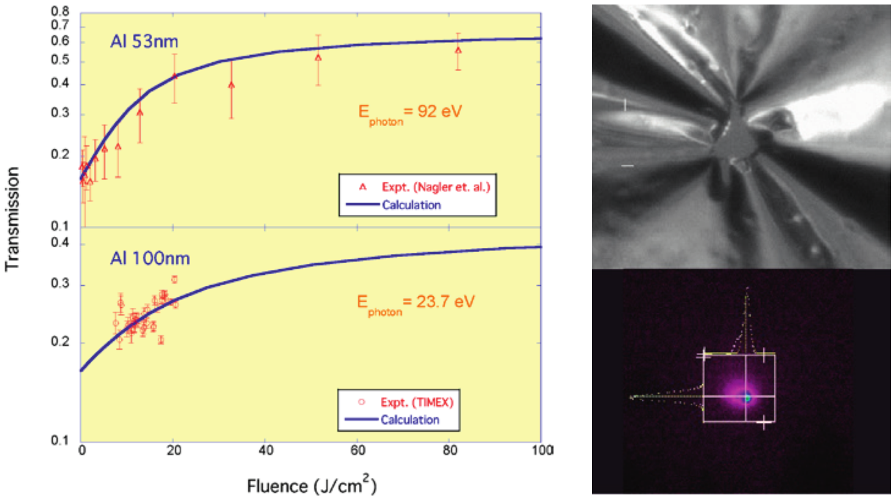

Figure 4

Left side: transmission of Al ultrathin foils as a function of the incident fluence of FEL pulses. The result of transmission measurements at the FLASH facility (see ref.[7]) and the first results obtained at TIMEX are compared with calculations (see text). Right side: the lower panel shows the lateral dimensions of the FEL pulses (10x10 µ FWHM) at focus (as observed on a YAG screen by the TIMEX telemicroscope), the upper panel shows the effect of about 100 repeated FEL shots (fluence 10-20 J/cm^2) on a 100 nm ultrathin Al foil. The pulses of seed laser were not filtered and concur to the damage of the foil.

Quite recently, we performed an experiment aimed at measuring the self-reflection intensity of a Ti sample (mirror) exposed to FEL pulses.[15] The Ti mirror (substrate: Si, roughness ~1 nm RMS) thickness 100 nm, passivated with 3 nm TiO$_2$) was loaded and aligned in the sample holder of the TIMEX experimental end-station. The FEL beam was focused onto the sample by the spherical platinum-coated silicon mirror placed close to normal incidence. The reflected intensity was collected at a 18.9 eV photon energy, at an incidence angle α = 6 degrees, by a Si photodiode (UVG20S, IRD inc) coupled with a 0.5 mm thick YAG fluorescence screen having a 100 nm aluminum coating (screening the laser seed optical signal) on the FEL side. The single-shot relative reflectivity variation $\Delta R/R$ was measured after careful calibration of the response function of the photodiode. We have also verified that the reflected intensity was not dependent on the particular region of the sample. The results are shown in Fig. 5 as a function of the incident fluence. Reflectivity data at low pulse fluence have been found to be practically constant within the uncertainty, as shown in Fig. 5 (left panel). The situation changes for high fluences, for which a clear increase well above the statistical uncertainty is found for fluence values greater than 5 J/cm^2 (see right panel of Fig. 5).

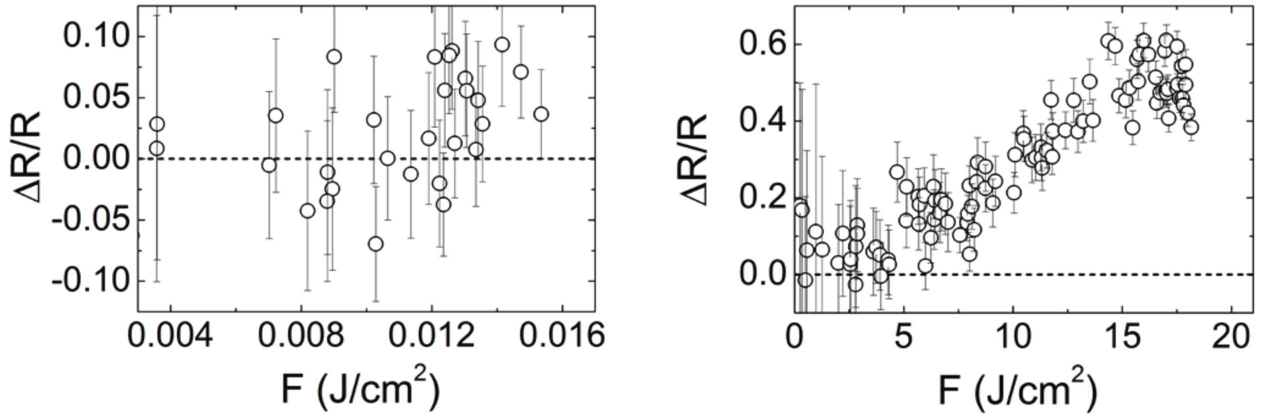

Figure 5

Normalized reflectivity change of a Ti mirror (roughness ~ 1 nm) as a function of the incoming pulse fluence (at 18.9 eV photon energy). The left figure shows that the reflectivity does not change within the estimated uncertainty for low fluence levels, while the right panel shows a marked increase above ~ 5 J/cm^2.

The reflectivity increase observed at high fluence is certainly an interesting phenonenon sheding light on the excited states reached during the excitation process. Its explanation is mainly associated with the excitation of electrons into a warm dense plasma within the pulse duration. A full account including the full set of data, details and interpretation of this experiment is given elsewhere.[15]

A second class of experiments exploits the tunability of the source and the excellent purity and energy resolution of the FEL pulses. In fact, FERMI@Elettra is a seeded machine, designed to deliver photon pulses with improved spectral stability and longitudinal coherence. Under those conditions, the possibility to scan the photon energy across an absorption edge of a given substance opens the way to measure EUV/x-ray absorption near-edge spectra (XANES) with the typical time resolution of the pulses (10-100 fs), following the dynamics of excitations through proper pump-probe schemes. The FEL frequency tuning scheme has been tested during the firstyearofactivityofthesourceand-detailsandresultsarepresentedelsewhere.[2] In particular, fine and coarse wavelength tuning of the FEL1 facility has been used to to scan across an atomic resonance (1s-4p resonance in He atoms, at 23.74 eV) and the Ge M4,5-edge of a thin Ge foil, respectively.[2]

A set of selected x-ray absorption measurements, carried out in a wavelength range including the Ge $M_{4,5-}$ edge, is shown in Fig. 6. For comparison, the theoretical absorption curve of a Ge foil (40 nm thickness) is superimposed on the experimental data points. The experimental data of this initial XANES experiment are in good agreement with the theoretical transmission profile, although experimental data are affected by a rather large error bar. The relatively large uncertainty of those preliminary data is associated with poor thickness homogeneity and the intrinsic fluctuations in the detection of the shot-by-shot I_0 (incident flux) and I_1 (transmitted flux). However, data reported in Fig. 6 show that ultrafast XANES spectra can be obtained at the FERMI@Elettra facility. Recent x-ray absorption measurements on ultrathin Ti foils,[16] measured under improved conditions, show the potential of this technique for investigating high energy density states of matter.

4. CONCLUSIONS AND PERSPECTIVES

The TIMEX end-station is operating at the FERMI@Elettra FEL facility and allows performance of experiments on transient and excited states of condensed matter. The present experimental set-up can be used with FEL1 radiation for investigating the EUV/x-ray absorption of ultrathin foils and the reflection of low-roughness surfaces (mirrors). The tunability of the FEL source has been exploited for EUV (x-ray) near-edge absorption spectroscopy (XANES) experiments. In this report we have briefly mentioned some preliminary results including: saturation effects for high fluence pulses; reflectivity change as a function of fluence; measurement of the near-edge spectrum near the Ge $M_{4,5}$ edge.

Many developments are in course of action or planned at the time of writing. A key development, planned to be completed before the end of the year, is related to the installation of an optical

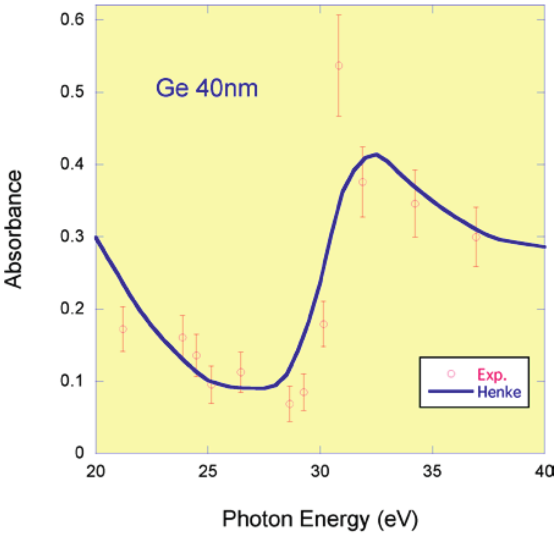

Figure 6

First near-edge $M_{4,5}$ x-ray absorption spectrum (dots with error bars) of a 40 nm Ge ultrathin foil collected at FERMI@Elettra,[2] compared with the calculated absorbance (blue line[17]).

jitter-free pump-probe set-up, as shown in Fig. 7. A fraction of FEL seed laser (780 nm), will be delivered to the TIMEX sample chamber, where it will be delayed (0-1 ns) and focused by a dedicated optical setup. Collection of ultrafast optical absorption and reflectivity data in single-shot pump-probe[3,13] experiments (see Figs. 7 and 1) at selected time delays are able to give important information about transient states. Fast CCD cameras and diode array detectors will be used for detection of optical ultrashort pulses within this pump-probe scheme. Another development effort will be devoted to the installation of a x-ray emission spectrometer,[12] object of a specific project involving three partners. A parallel effort will be devoted also to the necessary improvements of the detectors used for collecting the incoming, transmitted and reflected pulses. The final delivery and commissioning of the elliptic focusing mirror will be finally a major upgrade allowing for much better performances in terms of maximal fluence in the whole photon energy range of FEL1 and FEL2. In conjunction with the FEL delay line to be commissioned, it will also open the way to different experimental possibilities, like pump-probe experiments with the FEL first and third harmonics (see Fig. 7) also using FEL2 radiation.

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*Timex collaboration 2008-2012, University of Camerino and Sincrotrone Trieste, TIme-resolved studies of Matter under EXtreme and metastable conditions: http://gnxas.unicam.it/TIMEX

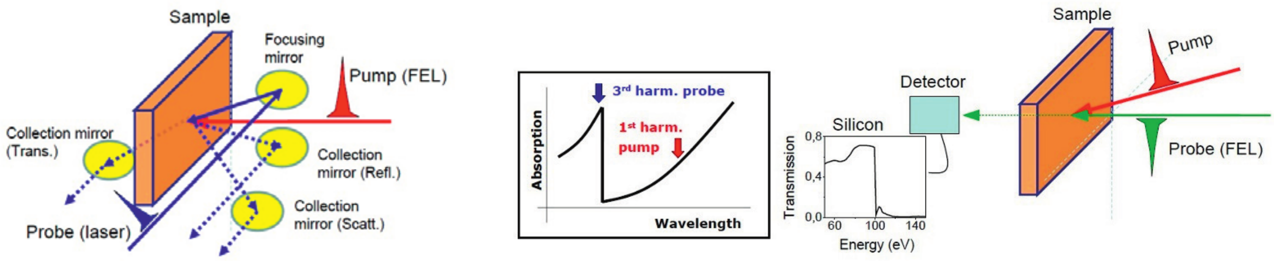

Figure 7

Sketch of possible pump-probe experiments at the TIMEX end-station, currently under development. In the left figure, a FEL-pump/optical-probe set-up with reflection/absorption data collection is sketched. In the right figure, a pump-probe experiment using 1st and 3rd harmonics of the FEL pulses is sketched (ultrathin Si as an example). Different pump-probe schemes using an optical pump are also possible.

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