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PREPARED FOR THE U.S. DEPARTMENT OF ENERGY,
UNDER CONTRACT DE-AC02-76-CHO-3073

PPPL-2752
UC-426

PPPL-2752

OVERVIEW OF THE PROGRAM ON SOFT X-RAY LASERS AND THEIR APPLICATIONS AT PRINCETON

BY

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May 1991

PRINCETON
PLASMA PHYSICS
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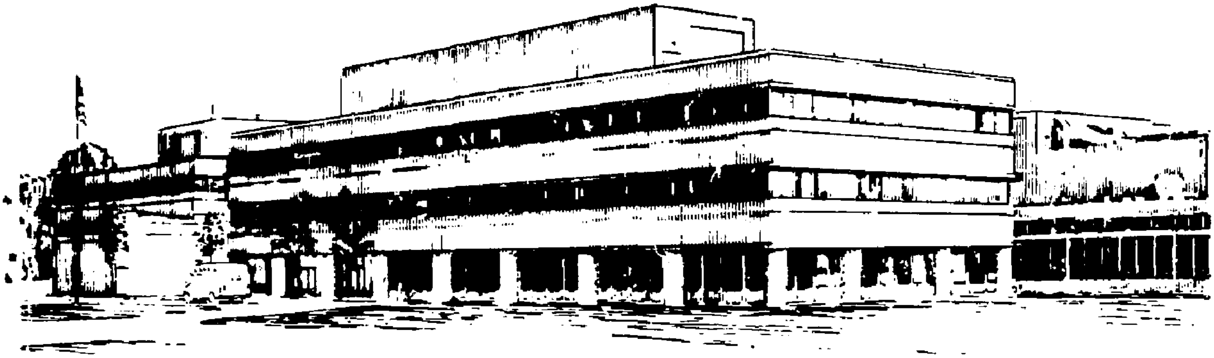

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OVERVIEW OF THE PROGRAM ON SOFT X-RAY LASERS AND THEIR APPLICATIONS AT PRINCETON

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ABSTRACT In the last several years, rapid progress in the development of soft X-ray lasers (SXL) has been observed at a number of laboratories worldwide. Although SXLs are very "young" devices they have already been used for microscopy and holography, and new ideas are emerging for broader application of SXLs to microscopy, holography and lithography. This paper describes the work at Princeton University on the development of a soft x-ray imaging transmission microscope using a SXL as a radiation source and work on the development of a novel soft x-ray reflection microscope and its application to biological cell studies and lithography. Progress in the development of a photopumped VUV laser (60 nm), and programs for the development of a small scale SXL and for the application of a powerful subpicosecond KrF laser system are also discussed.

1. INTRODUCTION

During the four years since the Aussois Conference (1986), rapid progress has been observed in many areas notably in (i) the development of soft x-ray lasers (SXLs) at shorter wavelengths down to the "water window" (Matthews 1990, MacGowan 1990), (ii) in the successful use of an SXL amplifier for the correction of refraction (Neely 1990), (iii) the first demonstration of a photopumped SXL (Boehly 1990), (iv) very elegant cavity experiments (Ceglio 1988), and (v) the first applications of SXLs to holography (Trebes 1987) and microscopy (DiCicco 1988, Skinner 1990). Very encouraging results in the development of small scale SXLs, have been reported (Kim 1989, Skinner 1990). Such "portable", relatively inexpensive SXLs might find application in many laboratories for the testing of optical elements, detectors and photoresists, and for optical alignment (e.g. in lithography), microscopy, and many others applications in the soft x-ray region. The importance of the development of small scale SXLs is motivating new ideas for pumping with lasers (Muendel and Hagelstein 1990) and capillary discharges (Rocca 1989, Lee 1990).

The development of relatively compact X-ray lasers at wavelengths in the 1 nm range, requires a very high pumping power density, about 3 to 4 orders of magnitude higher than for 10-20 nm lasers, and ultra-short pulse lasers (of picosecond and sub-picosecond duration) have been developed to this end (see e.g. Boyer 1988, Szatmari 1987, Watanabe 1988 and Nam 1990). These lasers, of size usually not exceeding 2-3 optical tables, can deliver focal power densities of about $10^{18}W/cm^2$. This creates opportunities for extremely interesting new research in addition to the search for efficient and not very expensive methods for the generation of lasing action in the wavelength region from 5 nm down to 1 nm.

In research for the development of SXLs at shorter and shorter wavelengths, the longer VUV wavelength region (from 100 nm down to 50 nm), so important for the applications in chemistry, is very often overlooked. Radiation at the wavelength of 20 nm and below is too energetic for chemical applications (the photon energy is significantly larger than the molecular bond energy). Some longer wavelength experiments such as the generation of lasing action near 100 nm in XeIII by Auger transition (Kapteyn 1986, Sher 1987) have good potential to be extended to much shorter wavelengths along isoelectronic sequences. A VUV experiment at Princeton is based on resonant photopumping of MoVII by MoXII. This was proposed approximately two years ago by Feldman and Reader (1989) for development of a VUV laser at ~60 nm, and may show the way to achieve a high repetition rate SXL.

In this overview we present works on the application of the SXL to microscopy, some ideas related to X-ray lithography, and recent experimental results on the development of a VUV laser in the vicinity of 60 nm. We will also describe plans for the development of a small scale SXL and for applications of the powerful sub-picosecond KrF laser system for high harmonic generation and for the study of the effects of the very high electromagnetic field created in the focus of this laser on the structure of materials.

II. APPLICATIONS OF THE SXL

1. Microscopy

The Princeton SXL at 18.2 nm, pumped by a 300J CO_2 laser, has sufficient beam energy and narrow divergence (Suckewer 1986) to expose photoresist in single shot. Photoresist is much less sensitive than film and requires an energy density of the order of $100 mJ/cm^2$ for exposure. The first application of the 18.2nm laser was for soft x-ray contact microscopy of biological specimens (DiCicco 1988 and Skinner 1990) The ultimate goal of our X-ray laser microscopy program is to obtain images of living cells. Very impressive images of cells have been obtained using soft x-rays from synchrotrons, however the exposure time is a fraction of a minute. The special advantage of the SXL is that the short pulse (of the order of nanoseconds) offers the potential of observing a cell that was alive the instant before the flash exposure from a SXL recorded its image. The necessary radiation dose levels make it unlikely that the cell will survive the exposure but exposures of several similar cells in identical conditions taken at certain time intervals should allow new information to be obtained about dynamic processes inside cells.

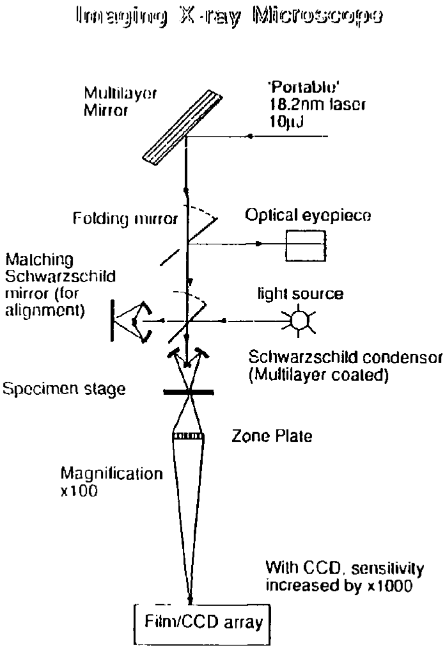
The "water window" region between the oxygen and carbon K edges (2.3 nm - 4.4 nm) is customarily regarded as ideal for X-ray microscopy, however at 18.2 nm the absorption cross section of oxygen is three times that of carbon providing good contrast between carbon in the cell proteins and the oxygen in the surrounding water. Although, up to now, most of our knowledge of the internal structure of cell has been learned from electron microscopy, there are questions about the fidelity of the image to the original living cell. In order for a cell to be observed in an electron microscope, the cell has to be stained with metals, dehydrated and sliced. It is obvious that some information about the living cell is lost or distorted in this process. In the soft x-ray region there is sufficient contrast between the different components of biological cells so that only minimal preparation is required. Of course a live cell can be viewed with an optical microscope with high fidelity but the resolution is much lower than with electron or x-ray microscopes.

In the x-ray laser contact microscope, a thin (0.1μ) silicon nitride square window (0.2 mm x 0.2 mm) separates the vacuum tube, in which x-rays travel, from the biological cells located on photoresist at atmospheric pressure. The shadow of the specimen printed on photoresist and magnification is obtained when the exposed photoresist is viewed in a scanning electron microscope (SEM). (The resolution of photoresist is the size of the polymer molecules ~5-10nm) Images of diatoms fragments (the silicified skeleton of planktonic algae) on photoresist indicated that the resolution on the photoresist was better than 0.1μ. One may also regard diatom fragments as a kind of lithographic mask (see e.g. Fig.3 in Skinner et al. 1990) and an illustration of the potential application of the SXL to microlithography, discussed in section 2.

In the application of x-ray microscopes to biological cells it is very important that such microscopes are practical, convenient to use and permit biologists to observe and manipulate cells under an optical microscope prior to creating a high-resolution image of these objects on photoresist with the SXL beam. We have developed such a microscope, which combines an inverted phase-contrast optical microscope with a soft X-ray laser contact microscope. It is called Composite Optical/Soft X-ray Laser Microscope "COXRALM", and is shown schematically in Skinner 1990. The inverted optical phase microscope has already been used as a microspectrofluorometer for the study of transient coenzyme fluorescence changes in living cells as a result of the intracellular microinjection of metabolites (Hirschberg 1979). This research is aimed at understanding the pathology and physiology of cancer cells, however, the inevitable resolution limitations of the optical microscope resulted in many features of the metabolic processes inside the cells remaining obscure.

COXRALM offers the possibility of making an X-ray contact image of these cells while preserving the ability to select and observe through the optical microscope and perform micro-injection of metabolites and/or inhibitors and xenobiotics just prior to X-ray exposure. In order to effectively use the optical part of the microscope the cells are mounted on resist on a transparent substrate. When a contact X-ray image is desired, the SXL beam tube is lowered until the silicon nitride window at the tip is in contact with the cell and the SXL is then triggered. A SEM image of a replica, produced by the 18.2 nm SXL, of dehydrated HeLa cells (Helen Lane cervical cancer cells) obtained in cooperation with the Biology departments of Princeton University and the University of Miami was presented in Suckewer and Skinner 1990. Presently our work is concentrated on experiments with live cells in a wet environment and on the development an Imaging Soft X-Ray Laser Microscope (IXRALM).

A magnification of a factor of 100 can be obtained in an imaging microscope, by using soft x-ray optics based on Schwarzschild objectives with multilayer coating and micro-zone plates. This allows the use of relatively low resolution film or CCD detectors which are about 10^6 times more sensitive than photoresist. Although losses in the optical components of IXRALM may decrease the photon density at the detector by a factor of 10^3, nevertheless the overall sensitivity of imaging microscope is expected to be $\sim 10^3$ times higher than the contact microscope. In the imaging microscope a small scale "portable" SXL of energy $10\mu J$ can replace the millijoule SXL used for contact microscopy. A schematic of IXRALM is presented in Fig. 1 and the first prototype, installed on SXL, is shown in Fig. 2. In IXRALM, a Schwarzschild objective is used as a condensor and has a numerical aperture (NA) matching the zone plate. The multilayer coating on Schwarzschild objective was provided by N. Ceglio's group (LLNL), and consists of 10 pairs of layers of Mo and Si each 9.25 nm thick. The wavelength of maximum reflectivity is shifted on convex mirror relative to concave mirror by 0.4nm and this makes the effective reflectivity at 18.2 nm about 10% from each surface. Also shown in the schematic is a second identical Schwarzschild objective for the interferometric alignment of the condensor and specimen (but not zone plate). In our initial experiments with IXRALM we have encounter difficulties with the very precise positioning required of the zone plate with respect to the specimen and condensor. Therefore recently we have reassembled our imaging systems with the zone plate replaced by a second Schwarzschild objective. Now all systems can be pre-aligned with a visible laser beam and the SXL beam



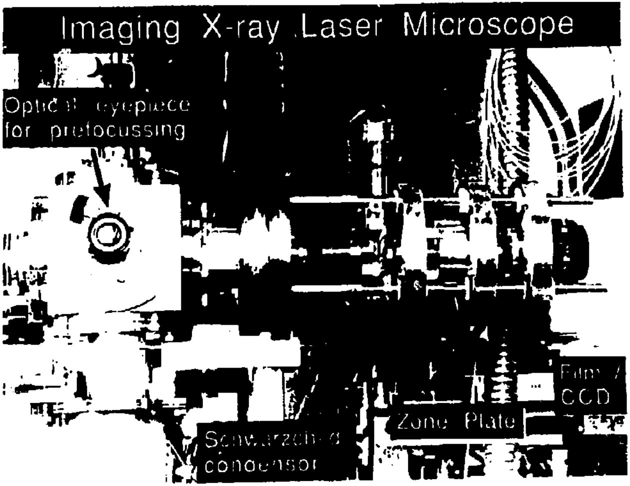


Fig. 2. Photograph of IXRALM on the X-ray laser

Fig. 1. Schematic diagram of IXRALM

is required only for the final adjustment of the system. Present work is aimed at the creation of a good image of the specimen in the detector plane.

2. Lithography

We consider at present three areas in the application of SXLs to X-ray lithography: (a) inspection of masks and integrated circuits (ICs) with high resolution holographic microscopy, (b) the SXL as a source of soft x-ray radiation in integrated circuit production, (c) alignment and testing of optical components.

This order of SXL applications represents in our view, the order of importance of the SXL for lithography. Let's start our discussion with the quite obvious and the simplest case (c). With progress in the development of small scale ("portable") SXLs and the expectation that such lasers with beam energy of order of micro-joules will soon be commercially available, the prospects for using small scale SXL's in the immediate future for alignment and testing of optical components primarily in projection X-ray lithography, seem to be very realistic.

The second case (b) is a much more difficult task. First of all probably the best use of a SXL would be projection X-ray lithography at wavelengths 18.2 nm (CVI) and 12.9 nm (SiXII). Projection lithography, which became possible recently due to the development of multilayer mirrors with reflectivities up to 50-60% in the region of 10-20nm region (Ceglio 1989), is gaining momentum and first results are very encouraging (Berreman 1990). However for the SXL to be considered as an appropriate radiation source for IC production via X-ray lithography, its average beam power should be in the range of 100-200 mW. This implies that SXL pulses of ~100mJ energy should be generated at 1-2 Hz repetition rate. This task seems to be feasible if the CO_2 laser pumping system in our SXL could be replaced by an appropriately constructed electrical discharge system and the pulsed magnet replaced by a steady state superconducting magnet. A special problem is the target design, which would be a modified version of the existing target for the CO_2 laser pumped SXL. In our opinion such an efficient and powerful SXL is possible for 18.2 nm, 15.4 nm and 12.9 nm wavelengths (the last wavelength is the most suitable for projection lithography).

Presently, the most novel lithographic application of the SXL seems to be in the area of relatively fast inspection of masks and integrated circuits (point a) at high resolution (~0.05-0.1μ). Of course, an electron microscope can provide very high resolution images, however the procedure is too slow and subject to damage by the energetic electron beam. Mask and IC inspection with soft x-ray radiation exploit two important features of the SXL beam: (a) significant reflectivity and contrast near 18 nm for number of materials (including biological specimens) for radiation non-normal to the surface (e.g. angles 30° and 45°; see Table 1) and (b) coherence. The system consists of two parts: a device for recording a high resolution hologram of an IC (or mask) and a reflection X-ray microscope (Suckewer 1989). The role of the X-ray hologram (interferogram) will be to localize defects by computational comparison of the hologram of the investigated IC with the hologram of standard (non-defective) IC, stored in the computer memory. Once areas with defects are localized, the X-ray reflection microscope will provide images of the defects at a resolution of 0.05-0.1μ. A large improvement of SXL beam coherence is expected with the achievement of multipass gain inside cavities. The motivation for using holography is that specialized techniques are needed for rapid defect identification in order to process the very large amount of information involved (e.g. there are 10^{10} 0.1μ pixels in a 1 cm^2 mask or IC). One possible configuration involves a coherent SXL beam which is incident at 45° onto both an IC (or mask) and a reflection zone plate. The zone plate, with Mo/Si multilayer coating (reflectivity > 30%), provides a spherical reference wavefront. This will interfere with the light scattered from the IC (or mask) to generate a Fourier transform hologram which can be recorded by a CCD and stored on a computer.

Table 1. Reflectivity of 18.2nm

Material	45 degree reflectivity %	30 degree reflectivity %
Au	3	14.3
C	0.5	3.5
Si*	0.04	0.09
SiO_2	0.2	1.5
Si_3N_4	0.3	2.0
Os	4.5	23.0

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*Si L edge is at 17nm hence the low reflectivity near this wavelength. All material bulk solid, assumed optically smooth, unpolarized illumination. Based on Henke data as interpreted by S. Mrowka of Oxford Research, Richmond CA.

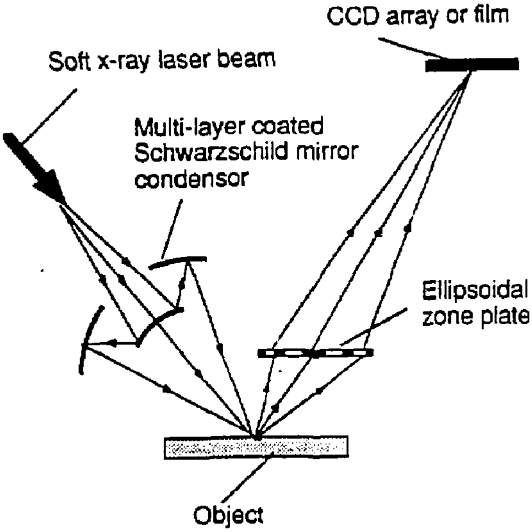

Fig. 3 Schematic of reflection microscope

Initially a hologram of a standard IC or mask (free of defects) should be recorded. Holograms recorded from subsequent ICs or masks will be compared to this standard and the difference reconstructed to provide the location of defects.

There are several issues here, such as the effectiveness of the holographic technique, the requirement of sufficient intensity and contrast in the scattered X-rays, the coherence of the SXL beam, and the reconstruction and interpretation of the hologram.

Once defective areas have been located, they can be inspected at 0.05-0.1μ resolution by a reflection imaging 18.2nm microscope, using a zone plate as the imaging lens. Because of the limited field of view, short focal length and very small depth of focus of zone plates, it will be advantageous to use an elliptical zone plate. The schematic of the reflection microscope is shown in Fig. 3. The SXL beam is directed at an angle (~45°) to the surface and is focussed on the object by a multilayer coated Schwarzschild condensor. Radiation reflected from the object enters the ellipsoidal zone plate which a provides magnified image of the object surface. The 18 nm wavelength is the most appropriate for such a system from the point of view of the reflectivity coefficient at 18 nm and the high resolution desired (at shorter wavelengths the reflectivity coefficient is smaller and at longer wavelengths resolution is lower). This reflection soft x-ray microscope can, of course, be used for the study of surfaces (e.g. membranes) of biological specimens such as, live cells with relatively good contrast between cell components due to significant differences in their reflection coefficients.

III. PHOTOPUMPING EXPERIMENTS FOR THE DEVELOPMENT OF A 60 NM LASER

In the race towards X-ray lasing at shorter and shorter wavelengths the very important VUV region near 60 nm has been often overlooked. A laser at this wavelength (photon energy ~20 eV) is ideal for various experiments in chemistry, for which the photon energy of SXLs is already too large. A VUV laser can, in principle, be operated at a high repetition rate, and this is often a necessary feature for the effective use of such lasers in chemistry.

In the search for an appropriate lasing scheme near 60 nm, the approach proposed by Feldman and Reader (1989) seems to be quite realistic. They pointed out that there was a unique line coincidence for two different ions of the same element, namely the Mo XII 5s $^2S_{1/2} \rightarrow 4p\ ^2P_{1/2}$ transition at 13.65 nm is coincident with the MoVII $4p^5\,6s \rightarrow 4p^5\,5p\ ^1S_0$ transition. Both lines are strong lines to the ground level (see the Grotian diagram in Fig. 4), and are therefore well suited to the photopumping scheme. The pumping line of MoXII originates in a more central part of the plasma where the temperature and density is higher than the outer regions where Mo VII is abundant. This photopumping can be used to create a population inversion and gain on the Mo VII $4p^5\,6s \rightarrow 4p^5\,5p$ transition in the vicinity of 60 nm.

Resonant photopumping for X-ray laser development was the first proposed by Vinogradov et. al (1975) and Norton and Peacock (1975). In the past, the shortest wavelength photopumped laser was near 200 nm. Very recently photopumping seems to be successful in generating lasing action near 30 nm (Boehly 1990).

In our approach towards the realization of the Feldman and Reader (1989) scheme we have been using a 10J, 150 nsec CO_2 laser pulse, split into two beams and focussed on the target with significantly different power densities for the generation of separate MoVII and MoXII plasmas ("cold" and "hot" plasmas). The experimental set up is shown in Fig. 5 and the target geometry used in the experiments is presented schematically in Fig.6. In order to obtain a sufficiently hot plasma for a high fractional abundance of Mo XII one beam was focused to a small spot. The second beam was more weakly focussed for the generation of a cooler plasma of MoVII ions. Radiation from a limited region in the "cold plasma" (Mo VII) was selected by a mask and recorded on a VUV spectrometer. A key point from both successful and unsuccessful photopumping experiments was good control over the cold plasma temperature and density for the maximization of the abundance of MoVII ions. A second concern was the distance between the hot and cold plasmas. The MoXII pump flux was stronger when the two plasmas were closer, however the contribution of collisional excitation was also larger. This made the photopumping effect less clear and, more importantly, increased the population not only of the upper but also the lower potential lasing levels.

In the most experiments, the grazing incidence VUV spectrometer was equipped with a 600 grooves/mm grating for the wavelength range 30-70 nm, however in several

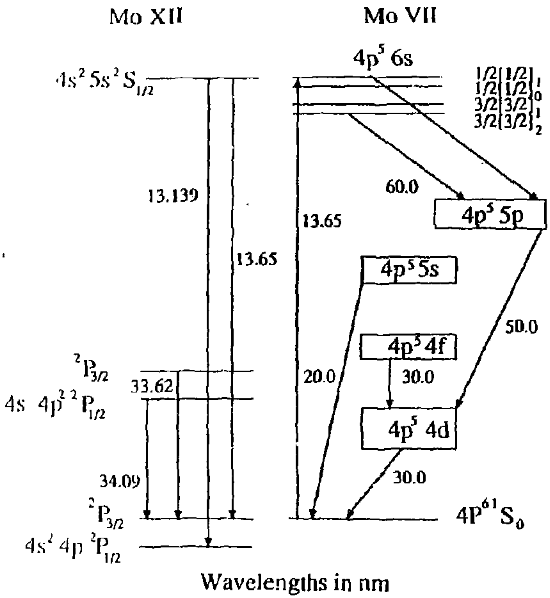

Fig. 4 Mo VII and Mo XII grotian diagram

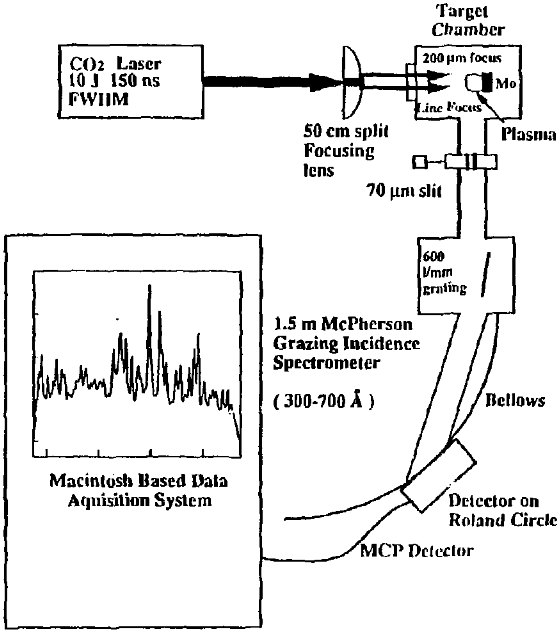

Fig. 5 Experimental Setup

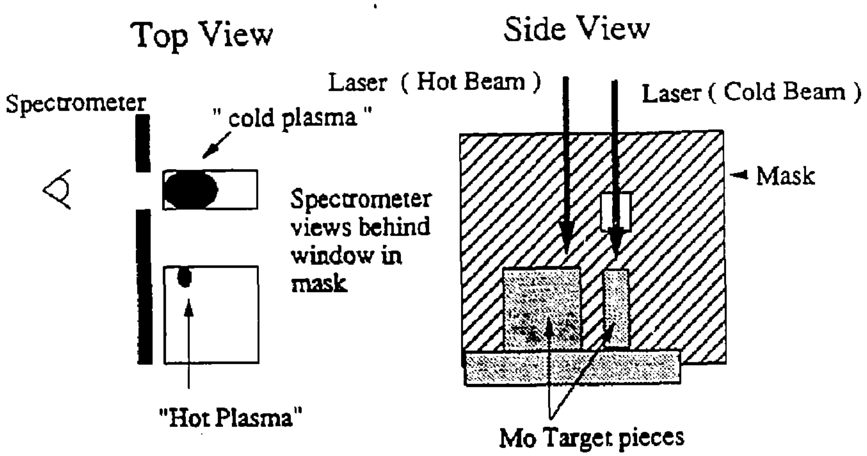

Fig. 6 Target Geometry

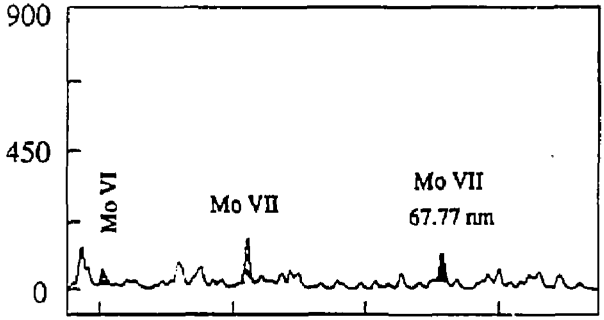

Fig. 7 a) Spectra from "cold plasma" only

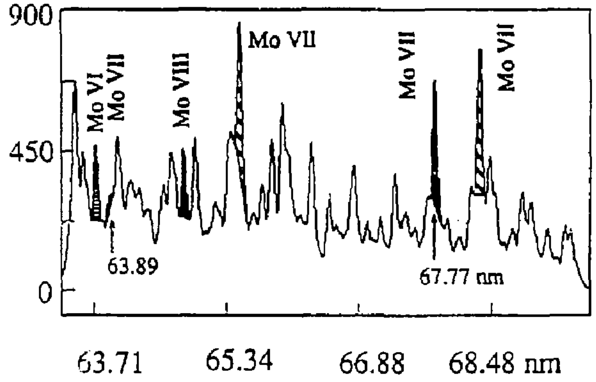

Fig. 7 b) Spectra from "cold plasma" pumped by Mo XII

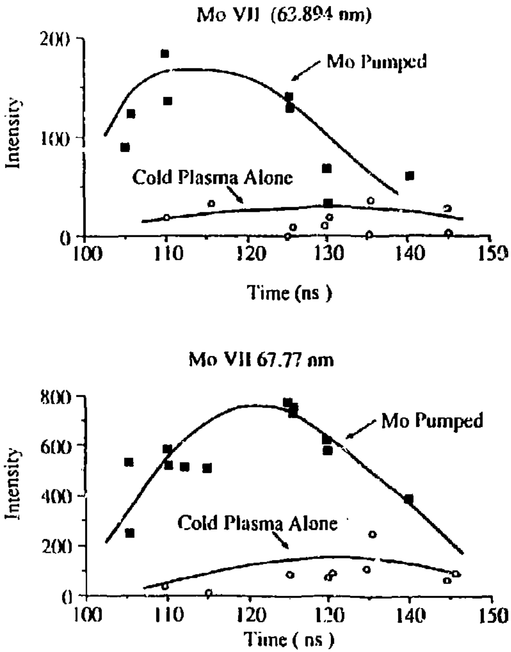

Fig. 8 Time Evolution of Mo VII potential lasing lines with and without photopumping

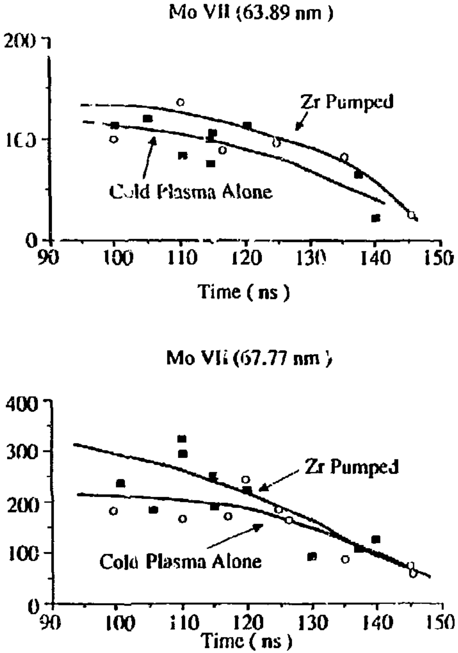

Fig. 9 Time evolution of Mo VII potential lasing lines when the hot plasma was created from a Zr (not Mo) Target

experiments a 1200grooves/mm grating was used. A multichannel microchannel plate detector registered a spectrum in one shot and the spectrum was displayed on a Macintosh PC. In more recent experiments a gating system was installed to obtain time-resolved spectra. The gating system operates in a variable (from 20 to 100 ns) window mode or in a mode in which the signal was integrated up to a selected time. In this second mode, the microchannel plate high voltage is clamped to ground at the selected time.

In the first series of experiments, by Ilcisin et al (1990) time integrated spectra in the vicinity of 30 nm and 60 nm were monitored during a scan of the experimental parameters in order to optimize the fractional abundance of Mo VII and Mo XII in the plasmas. With an appropriate selection of beam defocussing and expansion cooling suitable plasma conditions could be obtained.

In photopumping experiments, time dependent spectra in the 60 nm region were recorded for different target configurations. The most encouraging results were obtained for a Mo target consisting of 1 mm wide and 1 cm long blade located 1 mm from a second Mo XII target (see Fig. 6). The "cold plasma" with MoVII was generated on the 1 mm wide target by a CO_2 laser line focus ~1 cm long and the"hot plasma" (with MoXII ions) was generated by a smaller focal spot on the second target. Spectra from the "cold plasma" alone (Fig. 7b) and cold plasma pumped by "hot plasma" (Fig. 7a) shows strong enhancement of MoVII lines at the time corresponding the peak of the pump emission. The time evolution of two MoVII potential lasing lines (63.89 nm and 67.77 nm) with MoXII photopumping (upper curve) and without photopumping (lower curves) are shown in Fig. 8. When the Mo target was replaced by Zr in the "hot plasma" the enhancement of MoVII lines was very small (Fig. 9). This small enhancement was primarily due to collisional excitation from the interfering Zr plasma. In the Mo XII pumped plasma, enhancement of several other MoVII lines was also observed. Although transitions from the upper levels of these lines do not coincide with the MoXII 13.65 nm pumping lines, they lie below the $4p^5$ 6s levels of MoVII (upper lasing levels) and may be populated by collisional transitions from these levels.

In the near future, we plan to increase the photopumping rate by increasing the MoXII pump radiation intensity. This will be achieved by adding to the experiment a 5J (20 nsec pulse) ruby laser. We will also measure the population of the upper and lower lasing levels.

IV. SMALL SCALE ("PORTABLE") SXL

Work on the development of small scale SXLs is presented in detail by D. Kim et al. 1990). Therefore our discussion here will concentrate on some issues of more general character and our near future plans.

This work started as a part of the program on the improvement of SXL efficiency by the implementation of additional soft X-ray amplifiers and cavities (Suckewer 1988). Although we have succeeded in the generation of high gain/cm with relatively low pumping energy, we still need to increase the gain-length at least up to GL $\simeq$ 4.5-5 (up to now GL $\leq$ 3.3). Also improvement is needed in gain reproducibility. We have observed that gain fluctuates quite significantly from shot to shot, especially for very low pumping energy, and this makes it very difficult to conduct cavity experiments. We expect that an improved target design with cooling walls of high Z material will help in the generation of higher GL and the stabilization of gain. In the recent experiments with a Nd/Glass pumped small-scale SXL, we replaced the magnetic field by cooling walls to simplify the radial confinement of the plasma column. With gain of duration ~5-10 nsec several passes inside a cavity are achievable in such a SXL. This would dramatically improve the brightness and coherence of the laser beam. The reproducibility of the small scale SXL is a crucial factor in the development of a compact system for imaging microscopy.

In understanding the problems mentioned above 2-D, or even better 3-D, computer modeling would be very desirable. For the proper modeling we should, however, find the answer to the question why trapping of radiation from transitions depopulating the lower lasing level seems to be significantly smaller in recombining plasmas than predicted by calculations. Reliable measurements of the radiation trapping coefficients in plasma conditions similar to those used for generation high gain are needed.

V. APPLICATIONS OF THE POWERFUL SUB-PICOSECOND KrF LASER

Details of a powerful sub-picosecond (PSP) KrF laser and the complete system for the "Two-Laser Approach" to X-ray laser development were presented in Meixler, 1988 and Nam, 1990. Very recent results on the PSP-laser interaction with solid targets and plasmas have been discussed by W. Tighe et.al (1990). However, two aspects of our work with the PSP-laser outside of the program on X-ray laser development have not been previously reported. These are programs on the study of: (a) high harmonic generation, and (b) effects of very high EM fields (at the laser focus) on material (surface) structure.

Recently high harmonic generation (HHG) has been observed. Harmonics up to the 17th harmonic ($\lambda = 14.9$nm) of a ~0.25μ KrF laser (MacPherson 1987) and up to the 33rd harmonic ($\lambda \approx 32$nm) for ~1μ Nd/Glass laser (Ferray 1988) have been generated. Both showed quite a wide plateau region of harmonic radiation intensity vs. order of harmonics. The high harmonics were generated in the noble gases He, Ne, Ar, Kr and Xe. Our plan is to use our unique facilities to investigate the generation of high harmonics with the PSP-laser in ions of specific stages of ionization in a magnetically confined plasma created by a large energy (but relatively low power) CO_2 or Nd/YLF lasers. By selecting a proper delay between initiation of the laser generated plasma and the PSP-laser it is relatively easy to select an appropriate stage of ionization in the recombining plasma for the generation of high harmonics. The goals of the research are to optimize the efficiency of HHG, study the effects of various conditions on the size of plateau and to generate the shortest possible wavelength with a very high power density KrF laser ($\sim 10^{18}$W/cm^2). The system has been modified for this research and the experiments have begun.

Another use of the PSP-laser is to investigate changes in material structures at or near the surface caused by EM fields in the laser focus. At power densities $\sim 2\times10^{18}$W/cm^2 the electric field reaches $\sim 0.5 \times 10^{11}$V/cm (0.5 kV/Å). Such a field, is much stronger than the electric field in a hydrogen atom (~10V/Å), and significant transient and permanent structural changes in materials are expected. While permanent changes in the structure of materials could be investigated with a scanning electron microscope, observation of transient changes are a much more challenging task due to the short time scale of such processes. We plan to use a small portion of the PSP-laser beam as a probe of the material surface being exposed to the high laser field, using a beam reflection technique similar to the one previously developed by Milchberg et al. (1988).

ACKNOWLEDGEMENTS

We would like to thank the entire X-Ray Laser Division for dedication to the work, which was briefly presented here, especially D. DiCicco, D. Kim, K. Krushelnick, L. Meixler, D. Reiner, J. Robinson, R. Rosser, C. H. Skinner, W. Tighe, E. Valeo, and D. Voorhees. Encouragement and support provided for a number of years by H. Furth, R. Gajewski (DOE), and T. Stix were essential for all our achievements.

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