Contributed Paper

Development of Photoelectron Microscope with Compact X-Ray Source Generated by Line-Focused Laser Irradiation

YAMAGUCHI Naohiro[1], TAKAHASHI Zen[2], NISHIMURA Yasuhiko[2], SAKATA Atsusi[2], WATANABE Katsumi[3], OKAMOTO Yuuji[1], TAKEMURA Yuichiro[1], AZUMA Hirozumi[4] and HARA Tamio[1]

[1] *Toyota Technological Institute, 2-12-1 Hisakata, Tempaku, Nagoya 468-8511, Japan*
[2] *TOYOTA MACS Inc., Development Div. II, 2 Toyota-cho, Toyota 471-0826, Japan*
[3] *ULVAC-PHI Inc., 370 Enzo, Chigasaki 253-0084, Japan*
[4] *TOYOTA Central R&D Labs. Inc., 41-1 Yokomachi, Nagakute, Aichi 480-1192, Japan*

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A laboratory-sized x-ray photoelectron microscope was constructed using a compact x-ray source produced by line-focused laser irradiation. The system is a scanning type photoelectron microscope in which the x-ray beam is micro-focused via Schwarzschild optics. A compact laser-plasma x-ray source has been developed consisting of a YAG laser, a line-focus lens assembly, an Al tape-target driver and a debris prevention system. The 13.1 nm x-rays were delivered from the line plasma whose length was 0.6 to 11 mm with higher intensity than that from a point-focused source. Schwarzschild optics having a designed demagnification of 224 and coated with Mo/Si multilayers for 13.1 nm x-rays, was set on the beamline at a distance 1 m from the source. The electron energy analyzer was a spherical capacitor analyzer with a photoelectron image detection system that was suited for detection of a burst of photoelectrons excited by pulsed x-rays of ns-order duration. Photoelectron spectra were obtained having two peaks from As 3d and Ga 3d electrons, when a GaAs wafer was used as a sample. Spatial resolution of less than 5~3 μm was confirmed from the variation of the As 3d electron intensity along the position of the GaAs sample coated with a photo-resist test pattern.

Keywords:
X-ray photoelectron microscope, Laboratory-sized μ-XPS, Laser-plasma x-ray source, Line-focused laser, Schwarzschild optics

1. Introduction

Laser-produced plasmas have long been investigated as bright x-ray sources because of their high density and high temperature, and the availability of a variety of spectra by changing target materials [1,2]. Recently, laser-produced plasma x-ray sources have been highlighted as one of the most reliable sources for extreme ultraviolet lithography (EUVL). Intensive research has been performed in this field [3-5]. On the other hand, laser-plasma x-ray sources have been investigated for many other applications such as x-ray microscopy [6] and absorption spectroscopy [7,8]. In this paper a laser-plasma x-ray source is applied to an x-ray photoelectron microscope (μ-XPS).

X-ray photoelectron spectroscopy (XPS) is a useful technique for analyzing chemical composition and chemical states of surface materials. Recently, space-resolving XPS has been actively studied at many synchrotron radiation facilities [9-12]. A laboratory-sized μ-XPS system would be extremely useful for on-site inspection of products in various industries, especially in semiconductor fabrications. Laser-produced plasma x-ray sources have been used in laboratory scale μ-XPS systems [13,14]. Such laboratory-sized x-ray sources still do not provide sufficient photon flux to observe surface characteristics with high spatial resolution. Recently, an x-ray laser was applied for μ-XPS analysis [15], though the excitation laser was required to be highly intense and to have a rather precise pulse form for x-ray amplification. Utilizing non-lasing emissions in x-ray laser experimental geometry, an intense x-ray source can also be generated by line-focused laser irradiation with lower power density. This is because the x-ray intensity from a line-focused plasma is proportional to its plasma length, provided that the radiation is optically thin. Another merit of this source is that there would be a very few debris in the direction of the x-ray beam which is along the surface of the target [16].

We developed an x-ray source excited by line-focused laser irradiation with high repetition rate for practical use. The x-ray source was connected to a conventional photoelectron

author's e-mail: yamagch@toyota-ti.ac.jp
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energy analyzer via x-ray focusing optics. In this paper we report characteristics of the x-ray source and the performance of our μ-XPS system.

2. X-Ray Sources

Two types of x-ray sources were used in this work. One is a 10 Hz source that was used for the performance test in the preliminary stage. The other is a 50 Hz source dedicated for use in the μ-XPS system.

The 10 Hz source was excited by a pulse-train YAG laser whose wavelength was 1.064 μm. The pulse-train consisted of 16 pulses of 100 ps pulse duration with the interpulse time of 200 ps. Its energy was about 1.2 J, with over 90 % of the energy delivered into the first 8 pulses. The output laser beam was focused onto an Al tape target via a line-focus lens system with segmented prism array [17], providing an irradiation area of 50 μm × 11 mm. Because of interference in the focused beam, the irradiation pattern consisted of small dots. The power density was about 5×10^{11} W/cm^2 [18]. The tape target was made by sticking aluminum foil, 10 μm thick and 10 mm wide, on 0.1 mm-thick polyethylene-terephthalate tape, which was placed in the target chamber equipped with a tape driver and a debris shield system.

The soft x-ray emission from the 10 Hz source was observed by using a space-resolving flat-field spectrograph, consisting of a toroidal mirror, a grazing incidence spectrometer with an aberration-corrected concave grating (1,200 grooves/mm, Hitachi 001–0437), and a back-illuminated CCD camera (Princeton, SX-TE/CCD 512TKB).

A typical x-ray spectrum from the 10 Hz source is shown in Fig. 1. Prominent spectral lines are observed at around 13 nm, 15 nm and 16 nm. The cluster of lines around 15 nm originates from the transition lines of Al^{10+} ions, Li-like Al, which are well known as x-ray laser transitions [18]; therefore, it is necessary for a higher excitation laser power to produce these highly ionized ions. On the other hand, the 13 nm emissions are the transition lines of Al^{4+} ions and the 16 nm lines are those of Al^{3+} ions, and these x-ray emissions can be produced with the lower incident laser power. In this work the 13.1 nm line was used as a source for XPS analysis.

A cross sectional image of each spectral line viewed from one end of the line plasma can be reconstructed from the series of spectral data by changing the observation position along the target-normal direction. Figure 2 shows the spatial distributions of 13 nm and 15.5 nm lines. Though the source size of the 13 nm x-ray is larger than that of the 15.5 nm x-ray, its dimension is about 200 × 130 μm, which is small enough for microscopic applications.

The 50 Hz source was excited by a Q-switched YAG laser whose wavelength was 532 nm with a pulse width of 4–8 ns and nominal energy of 0.6 J. The output laser beam was focused via a combination of concave and cylindrical lenses into a line 0.6 to 1 mm long. The power density was estimated to be higher than 2×10^{11} W/cm^2. The dedicated target chamber contained a tape target driver using an open reel Al tape target and a debris prevention system with a

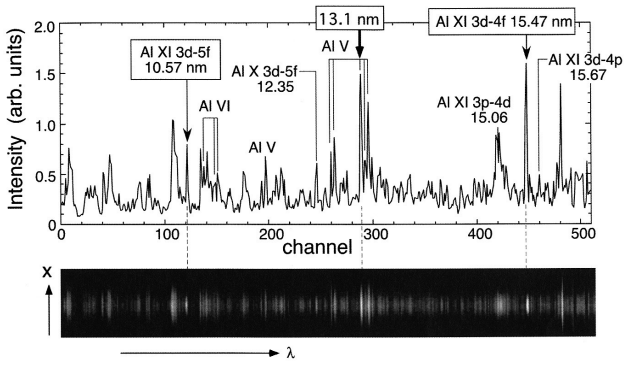

Fig. 1 Typical soft x-ray spectrum of Al plasma in the 10 Hz source.

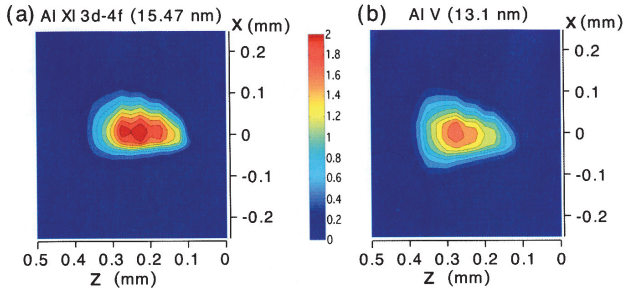

Fig. 2 Spatial distributions of 13 nm and 15.5 nm x-ray viewed from an end of the line plasma. The z-axis is along the target normal. The target surface is at z = 0 mm.

rotating quartz disc and rotating aperture disc [19]. Tape on the reel can be irradiated three times within its width and can be operated as long as 120 min without exchanging the reel.

The measured source size of the 13 nm x-ray was about 1.2 mm in the 50 Hz source. This is larger than that of the 10 Hz source. The x-ray source size could be approximately estimated from the product of the plasma front velocity and the duration of the excitation laser. The front velocity of the expanding plasma was measured to be ~(1–2) × 10^5 m/s. The estimated source size was 150–300 μm for the 10 Hz source and 0.8–1.6 mm for the 50 Hz source, which was in good agreement with the experimental results. In the 50 Hz source, a pinhole should be placed near the plasma to limit the x-ray source size for the present application.

A typical soft x-ray spectrum from the 50 Hz source is shown in Fig. 3, where the radiation is observed through a thin SiN filter; therefore, spectral lines appear only in the wavelength range longer than the Si L-edge, 12.5 nm. The source intensity has been estimated from the detected counts, N, in the spectroscopic measurement. The number of photons that pass through the incident slit of the spectrograph, N_{in}, is related to N by the following equation:

$$N = N_{in} \cdot \eta \cdot \varepsilon_{CCD} \frac{h\nu}{3.65} \cdot \frac{1}{g}, \qquad (1)$$

where η is the first order diffraction efficiency of the grating, ε_{CCD} is the quantum efficiency of the CCD detector, $h\nu$ is the

photon energy in eV, and g is the gain factor in the CCD camera system. The total number of emitted photons into 2π str. within ±2 % band width, N_{total}, can be related to N_{in} as

$$N_{\text{in}} = N_{\text{total}} \cdot \frac{\Omega}{2\pi} \cdot R \cdot T \cdot S \,, \qquad (2)$$

where Ω is the solid angle subtended by the toroidal mirror, R is the reflectivity of the mirror surface, T is the transmittance of the filter, and S is the ratio of the incident slit width to the size of source image on the slit.

The integrated count, N_{in}, for the 13.1 nm x-ray from the 50 Hz source was 3.4×10^6 in a single shot when an SiN filter was used and the toroidal mirror (30×60 mm^2) was set 800 mm distant from the source. By using $\eta = 0.04$ [20], $\varepsilon_{\text{CCD}} = 0.41$, $g = 5$, $T = 0.1$ and $R = 0.95$, the total photon number was estimated to be 2.6×10^{14} photons/shot·2π str.· ± 2 % BW, or 4 mJ/ shot·2π str.· ±2 % BW. This brightness would be sufficient for our applications.

3. μ-XPS System

The x-ray photoelectron microscopic system is shown schematically in Fig. 4. The sample chamber contains a Schwarzschild mirror, used to collect and focus the x-ray beam, a sample stage, and an electron energy analyzer. The Schwarzschild mirror (Olympus Optical Co.), coated with Mo/Si multilayers for the 13.1 nm x-ray, was set on the beamline at a distance of about 1 m from the x-ray source. The designed degree of demagnification was 224 on the image plane, as confirmed in the previous micro-beam experiment [21]. Attached to the chamber was also an electron gun for the alignment of the analyzer and an Ar ion gun for treatment of a sample surface.

Samples were set on a 4-axis manipulator with a high-precision piezo x-y stage (Physik Instrumente, Model P762-20). The electron analyzer (ULVAC-PHI, Model 1,600C) was a spherical capacitor analyzer with a mean diameter of 279.4 mm. The electron detector was an assembly of microchannel plates (MCPs) (Hamamatsu, F1208-01, tandem type) with a phosphor screen 30 mm in effective diameter. An image of the photoelectron spectrum was recorded and analyzed by using a very low dark-noise CCD camera (Hamamatsu, ORCA II-ER) and a personal computer. This system is suited for detection of a burst of photoelectrons emitted by a short x-ray pulse of ns-order duration. The energy resolution is limited by the size of a bright spot on the phosphor screen, estimated at 0.1 to 0.2 eV.

A GaAs wafer sample was analyzed. Because x-ray energy in our μ-XPS system is 94.6 eV, the outermost-bound electrons in the M-shell of the Ga or As atom would be excited. Ga 3d and As 3d photoelectron peaks were observed in the photoelectron spectrum image taken over 1–4 min accumulation for the 10 Hz source or 10 min for the 50 Hz source. A typical image is shown in Fig. 5. The intensity profile of the photoelectron image is plotted as a function of the kinetic energy of the photoelectrons in Fig. 6. Two peaks can be clearly seen in these figures. The peak at the lower

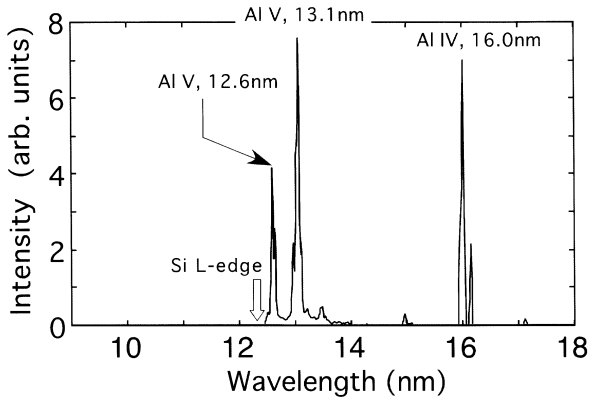

Fig. 3 Typical soft x-ray spectrum of Al plasma in the 50 Hz source. The spectrum was taken through a SiN filter.

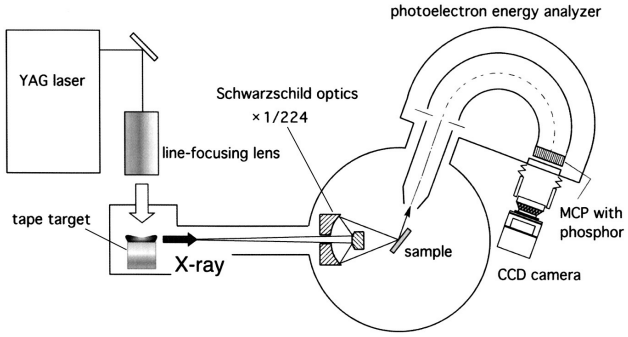

Fig. 4 Schematic layout of the x-ray photoelectron microscope using a line-focused laser-plasma source.

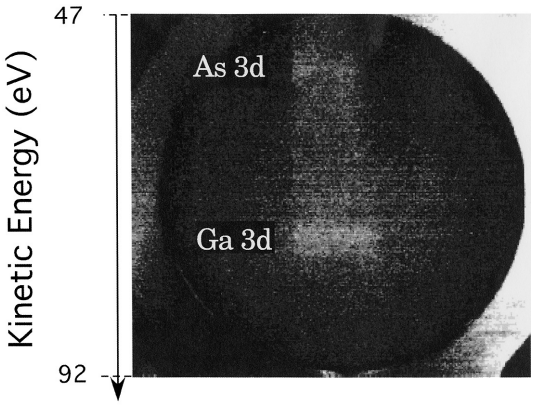

Fig. 5 Example of photoelectron spectrum image for a GaAs sample. Accumulation time was 10 min by using the 50 Hz source.

kinetic energy is assigned as the As 3d electron peak and the other one as the Ga 3d electron. The energy difference in the binding energy is measured to be 21 eV. The energy difference calculated from the tabulated values is 23 eV for elemental Ga and As substances or 21.2 eV for the GaAs compound [22]. Therefore, it is considered that these photoelectrons originate from atoms in the GaAs compound.

For the spatial resolution test, we used a GaAs wafer coated with photo resist, having a line and space pattern with a 40 μm period. The sample was moved precisely in the

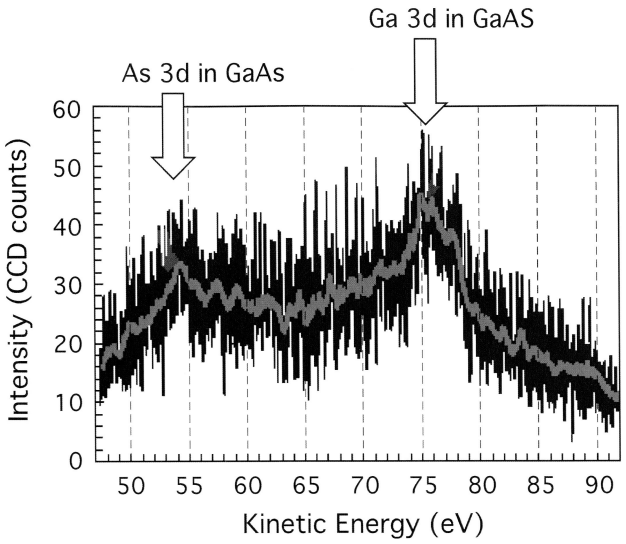

Fig. 6 Photoelectron spectrum for the image shown in Fig. 5. Two clear peaks are seen, which correspond to As 3d and Ga 3d photoelectrons.

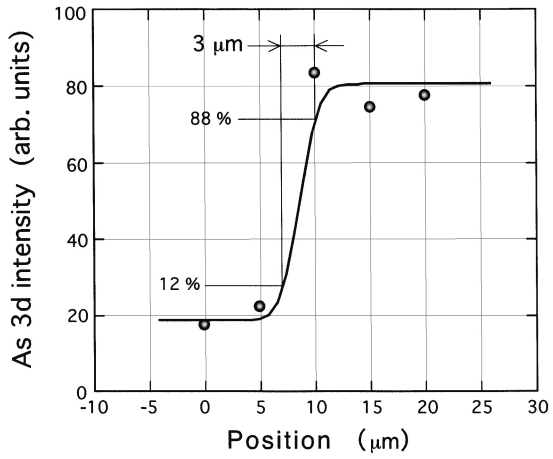

Fig. 7 Peak intensity of As 3d electron as a function of the sample displacement. The fitted curve, error function, is also shown. The spatial resolution is determined to be about 3 μm.

vertical direction to the patterned lines. Photoelectron spectra were obtained for each position. The peak intensity around the As 3d electron is plotted as a function of the sample displacement in Fig. 7 at the boundary of photo resist and the GaAs. Assuming the photoelectron intensity is proportional to the intensity of the probe x-ray and that the x-ray beam has a Gaussian intensity distribution, the data could be fitted to an error function. The best-fitted curve is shown in Fig. 7, from which the spatial resolution could be determined to be less than 3 μm. Because the distance between the mirror and the sample has not been optimized, the spatial resolution could be improved through further investigations.

4. Conclusions

Laser-plasma x-ray sources derived from irradiating a line-focused laser have been investigated for an x-ray photoelectron microscope. A 50 Hz source of 13 nm x-rays has been developed as a dedicated source, emitting an average intensity of 10^{16} photons/sec·2π str.·±2 % BW. A laboratory-sized μ-XPS system has been constructed using the line-focused x-ray source and Schwarzschild optics.

Photoelectron peaks due to the As 3d and Ga 3d electrons were observed when a GaAs wafer was used as a sample. The chemical states of As and Ga could be analyzed using this μ-XPS system. A spatial resolution of less than 5–3 μm was attained in this work.

Efforts should be continued to obtain sub-micron resolution by repeating adjustments of the x-ray mirror. We will also try to take two-dimensional images of energy-selected photoelectrons from an appropriate sample.

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