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Development of an X-ray photoelectron microscopic system with a compact X-ray source

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Abstract

We have constructed an X-ray photoelectron microscopic system. An X-ray source is a laser-produced plasma in a scheme of an X-ray laser experiment. X rays involving amplified spontaneous emissions (ASE) at 15.47 nm were delivered with a 10-Hz repetition rate from a compact X-ray laser system. X rays were collected and focused by a Schwarzschild optics coated with Mo/Si multilayers for a 15.47-nm X ray. Photoelectron signals due to the Ga $3d$ and As $3d$ electrons were observed, when a GaAs wafer was used as a sample. The spatial resolution of about 1 μm was confirmed.

Keywords: Microbeam; Schwarzschild optics; X-ray laser; X-ray photoelectron microscope

1. INTRODUCTION

X-ray photoelectron spectroscopy (XPS) is a useful technique to analyze chemical composition and chemical states of surface materials. Recently space-resolving XPS has been actively studied at many synchrotron radiation facilities (Ade *et al.*, 1992; Ng *et al.*, 1994; Johansson, 1997; Voss, 1997). In laboratory scale, an X-ray tube or a laser-produced plasma X-ray source is used as an X-ray source (Aoki *et al.*, 1993; Ohchi *et al.*, 1996; Kondo *et al.*, 1998). Such laboratory-sized X-ray sources still do not provide enough photon flux to observe surface characteristics with high spatial resolution. X-ray lasers are one of the X-ray sources which could realize microscopic XPS analysis. Because X-ray lasers have high brilliance, narrow spectral width, and small divergence, an XPS with such an X-ray source would be able to analyze materials with high spatial and spectral resolving powers.

We have developed a tabletop X-ray laser pumped by an yttrium aluminum garnet (YAG) laser. To realize such an X-ray laser, we proposed a pumping method using pulse-train lasers (Hara *et al.*, 1991; Hirose *et al.*, 1993). The use of the pulse-train laser is an effective method to obtain high-gain media of an X-ray laser through recombination processes. In our recent experiment, we observed amplifications of the two Li-like Al lines (10.57 nm and 15.47 nm) using the pulse-train YAG laser with an input energy of only 1.5–2 J/cm (Yamaguchi *et al.*, 1997) focused via a segmented lens system (Yamaguchi *et al.*, 1999*a*). The gain-length product for the $3d$–$4f$ transition of Li-like Al ions at 15.47 nm was about 3.5 and maintained for about 1 ns. Besides this X-ray laser output, we can utilize spontaneous emission from a line X-ray source, that is, not a point source. The X-ray output from the line source is thought to be intense enough to apply for an X-ray photoelectron microscope and has not been used for any micro-XPS systems yet. In this paper, we describe our X-ray photoelectron microscopic system with a line X-ray source involving an X-ray laser.

2. XPS SYSTEM

The X-ray photoelectron microscopic system is schematically shown in Figure 1. The pulse-train YAG laser whose wavelength was 1064 nm was focused onto an Al tape target, which was made by sticking aluminum foil 10 mm wide and 10 μm thick on a polyethylene-terephthalate tape of

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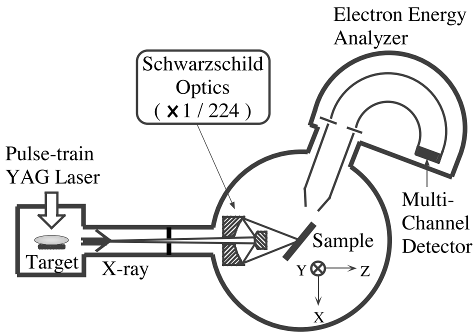

Fig. 1. Schematic diagram of the X-ray photoelectron microscopic system with a compact X-ray laser source.

0.1 mm thickness. By use of the tape target, an operation with a 10-Hz repetition rate was possible in our X-ray laser system. The focal line was 10 mm long and 50 μm wide. The shaped pulse-train consisted of 16 pulses of 100-ps pulse width with the interpulse time of 200 ps. Control of the envelope of the pulse-train leads to enhanced gain of soft X-ray lines and prolongs its duration in the recombination plasma scheme. The envelope of the pulse-train was shaped so that the former eight pulses are more than four times as intense as the latter ones (Yamaguchi *et al.*, 1997).

In a high repetition rate operation, it is necessary to protect optical components in the chamber from deposition of target debris. We devised a debris shield made of polyethylene film 75 μm thick and its feed mechanism (Hisada *et al.*, 1998). The debris shield was set in front of the target. It prevents contamination of an entrance window that the incident laser passes through. On the other hand, we have not prepared a special debris protector for the cavity mirrors near the target yet. Then X-ray laser was delivered from a target configuration without cavity mirrors in this work. X rays involving amplified spontaneous emission (ASE) at 15.47 nm in Li-like Al ion transition were emitted along the axis of a line plasma. The continuous operation duration of our X-ray source system is 30–40 min, that is limited due to the length of debris shield rolled on a bobbin.

The X-ray optical system used to collect and focus the X-ray beam on a sample surface is a Schwarzschild-type optics. The Schwarzschild optics consisting of a concave mirror and a convex mirror was set on the beamline at a distance of about 1 m from the X-ray source. X-ray reflection of such mirrors in near-normal incidence is enabled by using a multilayer structure. These mirrors were coated with Mo/Si multilayers for 15.47 nm X-ray. The measured reflectivity of the multilayer mirror was about 50% at the incident angle of 8°. The designed degree of demagnification is 224 on the image plane.

Samples were set on a four-axis manipulator with a high-precision piezo stage (Physik Instrumente GmbH & Co., Model P762-20). The electron energy analyzer (ULVAC-PHI Inc., Model 1600C) was a spherical capacitor analyzer with mean diameter of 279.4 mm. The electron detector was a microchannel plate (MCP) with 16 anodes. The energy resolution is about 50 meV in minimum.

The sample chamber contains the Schwarzschild optics and the sample stage, and is connected to the electron energy analyzer. An electron gun for Auger analysis and an Ar ion gun for treatment of a sample surface are also attached to the sample chamber. The chamber was usually evacuated by an ion pump to 1–3 × 10^{-7} Pa. When an X-ray beam was fed to the sample chamber, some pressure rise was observed but it was maintained at the 10^{-6} Pa range during XPS measurement.

3. EXPERIMENTAL RESULTS

Before constructing the XPS system, we performed a measurement of the size of the microbeam using the knife-edge method (Ohchi *et al.*, 1999). X-ray images through the knife-edge were detected by a two-stage MCP with phosphor screen of 53×53 mm^2 effective area (Hamamatsu, F2814-23P). visible images on the screen were recorded by a CCD camera (Hamamatsu, C4880). the measured characteristic curve of the knife-edge response at the best focus condition is shown in Figure 2. The dashed curve represents the fitting curve of the raw data to the error function and the solid curve shows its derivative. The beam size, which was the full width at the half maximum of this solid curve, was thus determined as about 0.42 μm. The photon flux on this spot were estimated from the result of the X-ray laser experiment; the photon flux from the X-rays involving ASE at the wavelength of 15.47 nm is measured to be about 6×10^{12} photons/shot/sr (Yamaguchi *et al.*, 1999*b*). The X-ray flux was reduced by one order less than this value when the cavity was not used. Taking into account the acceptance solid angle (3.9×10^{-6} sr) and the reflectivity of the Schwarzschild optics, we can estimate the flux in the spot to be about 2×10^5 photons/shot.

A GaAs wafer was used as a sample. Photoelectrons from the bound state of Ga 3*d* and As 3*d* levels in GaAs were observed in the photoelectron spectrum as shown in Figure 3. These data were obtained as the averaged data over

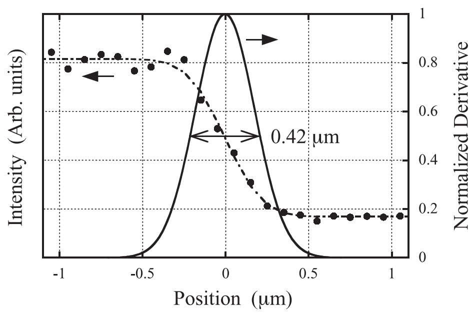

Fig. 2. Experimental result of the knife-edge test. The raw data obtained at each knife-edge position are shown by closed circles. The dashed curve is the curve fitted to the error function. The solid curve shows its derivative, which indicates the intensity profile of the microbeam.

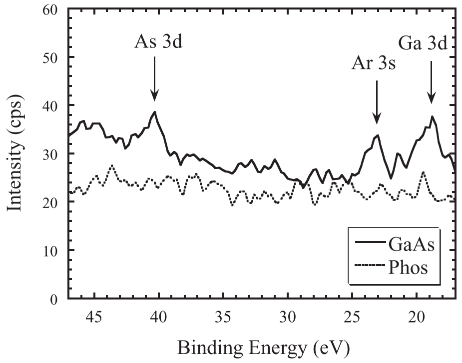

Fig. 3. Typical photoelectron spectra obtained by using this system. Solid trace shows the photoelectron spectrum for a GaAs wafer sample. That of a phosphor sample is shown by the dashed trace as a reference.

the 17 sets of measurements with about 7 min of total acquisition time. In the spectrum, the Ar 3*s* electrons were also observed, because the Ar ion gun was used to sputter the sample surface before the XPS measurement. In Figure 3 the photoelectron spectrum of a phosphor plate is shown as a reference, where Ar ion sputtering was not made. It is clear that this XPS system can identify chemical compound of a sample, though the S/N ratio is not enough in the present stage.

For a spatial resolution test, we used a photo-resist-coated GaAs wafer, whose pattern was a 20 μm × 20 μm lattice with a 20 μm-width gap. When the sample was moved along one direction, the variation of the intensity of photoelectron spectrum around the Ga 3*d* electron at each position was observed. The measurements were repeated by changing the position of the Schwarzschild optics along its optical axis, that is, by changing the spot size of the X-ray beam on the sample. It was observed that the modulation in the Ga 3*d* electron intensity was consistent with the pitch of the resist pattern on the sample for the larger X-ray spot. The peak intensity around the Ga 3*d* electron is plotted as a function of the sample displacement in Figure 4 for the smallest X-ray spot in this series of experiments. It can be considered that the four data points at low Ga 3*d* intensity in Figure 4 correspond to those when the X-ray microbeam is irradiated on the resist part. The sample displacement step was 6.25 μm. Therefore we can estimate the spot size, the spatial resolution, to be about 1–2 μm. However, it is expected that better spatial resolution would be achieved through more precise adjustment of the Schwarzschild optics.

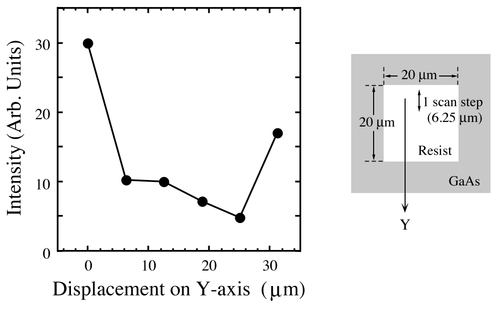

Fig. 4. Dependence of the intensity for Ga 3*d* electrons on the displacement of the sample. A schematic diagram of the resist pattern on the GaAs wafer is shown on the right.

4. SUMMARY

We have constructed an X-ray photoelectron microscopic system with a compact X-ray source. We equipped a microfocus beamline using a Schwarzschild optics. In the microbeam experiment, the focusing of the X-ray beam into a spot of about 0.42 μm in diameter has been achieved. The photon flux on the spot was estimated to be about 2×10^5 photons/shot. Photoelectron spectra from 17 eV to 47 eV in binding energy were obtained, when X-ray pulses involving ASE X-rays were delivered with a 10-Hz repetition rate. Photoelectron signals due to the Ga 3*d* and As 3*d* electrons were observed, when a GaAs wafer was used as a sample. The spatial resolution of about 1–2 μm was confirmed. Efforts should be continued to obtain a submicron resolution by repeating adjustments and measurements. After some improvements on the X-ray source system to permit a long continuous operation, we will try to take two-dimensional images of energy analyzed photoelectrons from this test sample.

The advantage of the use of a pulse X-ray source is that the photon flux comes into a sample in the very short time. It is possible to obtain the information of the instantaneous state of the sample surface by a single-shot irradiation. But at the present status, the output power is still not enough for various applications. We are planning to extract significant X-ray laser power by using more efficient cavity geometry, increasing the plasma length, and optimizing X-ray lasant conditions. So by improving the performance of the X-ray laser and by introducing a detection system suitable for photoelectrons coming into a very short period, we could expect one-shot detection of photoelectrons from a microscopic area.

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