

RESEARCH ARTICLE | JANUARY 15 2019

# Mo-overcoated grating-based beam intensity monitor for 13.9 nm x-ray laser **FREE**

Takashi Imazono ; Hiroaki Nishihara; Ryuichi Ukita; Hiroyuki Sasai; Tetsuya Nagano



AIP Conf. Proc. 2054, 060050 (2019)

<https://doi.org/10.1063/1.5084681>



CrossMark

## Articles You May Be Interested In

Experimental evaluation of enhancement of diffraction efficiency by overcoating diamond-like carbon (DLC) on soft x-ray laminar-type gratings

*AIP Conference Proceedings* (July 2016)

Germanium-overcoated niobium Dayem bridges

*Appl. Phys. Lett.* (August 2008)

Photoconductive properties of chemically deposited PbS with dielectric overcoatings

*Journal of Applied Physics* (September 2008)

500 kHz or 8.5 GHz?  
And all the ranges in between.

Lock-in Amplifiers for your periodic signal measurements



Find out more



# Mo-Overcoated Grating-Based Beam Intensity Monitor for 13.9 nm X-Ray Laser

Takashi Imazono,<sup>1, a)</sup> Hiroaki Nishihara,<sup>2)</sup> Ryuichi Ukita,<sup>2)</sup>  
Hiroyuki Sasai,<sup>2)</sup> and Tetsuya Nagano<sup>2)</sup>

<sup>1</sup>*National Institutes for Quantum and Radiological Science and Technology (QST), 8-1-7 Umemidai, Kizugawa, Kyoto 619-0215, Japan*

<sup>2</sup>*Shimadzu Corp., 1 Nishinokyo-Kuwabara-cho, Nakagyo-ku, Kyoto 604-8511, Japan*

<sup>a)</sup>Corresponding author: imazono.takashi@qst.go.jp

**Abstract.** For the purpose of developing a high-efficiency beam intensity monitor with a diffraction grating for a 13.9 nm Ni-like Ag X-ray laser (XRL), a molybdenum thin film was overcoated on a gold-coated holographic grating. The performance was examined by measurements of absolute diffraction efficiencies with synchrotron radiation, and the improvement effect by Mo-overcoating was confirmed. In addition, it became clear that a high correlation was obtained between the intensities of the zeroth and first order diffracted lights of the Mo grating measured with the XRL. To validate the practicality of this beam intensity monitor, the polarization state of the XRL beam of the zeroth order diffracted light delivered from the Mo grating was determined quantitatively by using rotating-analyzer ellipsometry with a Mo/Si multilayer polarizer.

## INTRODUCTION

Laser-produced plasma X-ray lasers (XRLs) have been developed worldwide [1]. The nickel-like silver XRL facility at the QST provides 13.9 nm coherent radiations with a 7 ps pulse duration generated by a double gain medium working as an oscillator and amplifier [2], and has been utilized for static and dynamic observations of laser ablation with materials [3,4]. In such experiments, it is important to accurately measure the number of photons per pulse in every incoming beam shot because of the XRL beam with a large intensity fluctuation mainly depending on the pumping laser and the excitation condition of plasma [1].

Amplitude- and wavefront-division beam splitting techniques, such as freestanding and membrane-supported multilayer films [5] and reflection mirrors [6], have been used as beam intensity monitors for soft X-rays. Amplitude-division beam splitters are challenging to implement and have several issues, such as the lack of rigidity, absorption and roughness of the membrane, and wavelength-selectivity. On the contrary, wavefront-division beam splitters have drawbacks that strongly affect the susceptibility from the intensity distribution and pointing stability of an incoming beam. As another method, gas detectors based on the photoionization of rare gases have a wide dynamic range and are effectively used below 300 eV except for resonance energies of the target gas [7]. Taking into account the easiness of the optical axis adjustment, it is important that such the beam splitters do not change the beam direction.

We succeeded in developing a Mo/Si multilayer-coated photodiode-based beam intensity monitor for the 13.9 nm XRL, being an s-polarization reflectance of 0.525 at an angle of incidence of 45° [8]. The beam intensity monitor generates the photodiode current proportional to the incident beam intensity as well as reflects the remaining light for downstream experiments. Therefore, it avoids the drawbacks of multilayer beam splitters, whereas the direction of the optical axis has been changed by reflection.

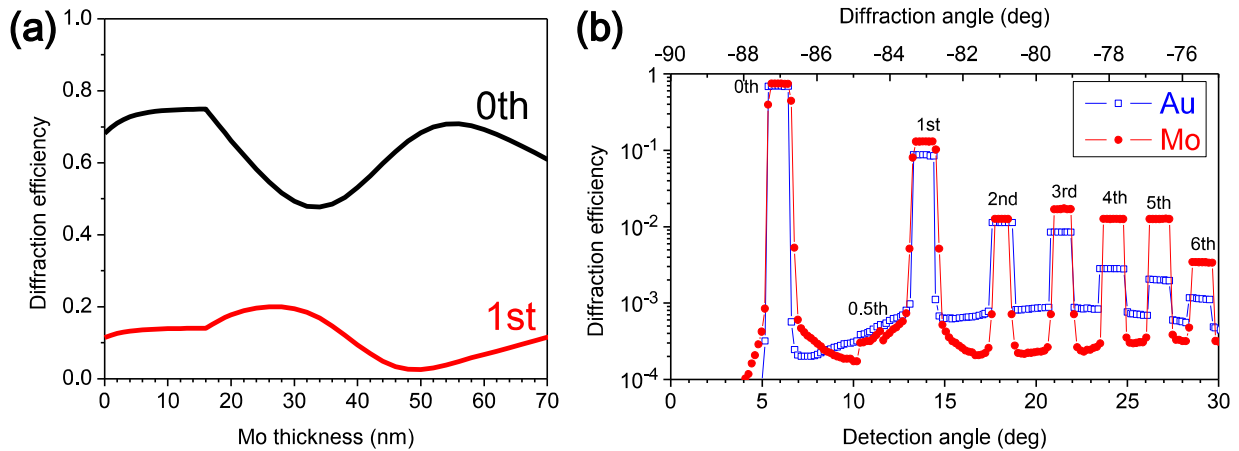
Transmission-type diffraction gratings made of diamond are used for monitoring the intensity of X-ray free electron laser (XFEL) [9]. On the contrary, in soft X-rays below 1 keV, reflection-type gratings are promising. Recently, it has been confirmed that high-density amorphous carbon such as diamond-like carbon (DLC) overcoated

on a gold-coated holographic grating has improved the diffraction efficiency [10]. Although a reflection grating-based beam intensity monitor as well as the multilayer-coated photodiode-based one changes the direction of the light path, it is likely to be superior from the viewpoint of the throughput and no wavelength-selectivity [8].

For the purpose of developing a grating-based beam intensity monitor, we propose a gold-coated holographic grating overcoated with a thin molybdenum layer to improve the diffraction efficiency at 13.9 nm. We report the performance of the Mo-coated grating with synchrotron radiation and XRL sources as well as validate the practicality of the grating-based beam intensity monitor through ellipsometry measurements performed with the XRL beam delivered from the grating.

## DIFFRACTION EFFICIENCY IMPROVEMENT BY MOLYBDENUM OVERCOATING

The grooves of replica gratings are formed in epoxy resin, whose surface is typically coated with a thin gold film that plays roles as a reflector as well as a parting agent from the master grating. The larger the real part of the complex refractive index is and/or the smaller the imaginary part (extinction coefficient) is, the higher the reflectance of the single layer film becomes. The complex refractive indices of Au and Mo are  $0.894 + i0.060$  and  $0.917 + i0.007$  at a wavelength of 13.9 nm, respectively [11]. It is expected that the reflectance of Mo is higher than that of Au owing to the large difference in the imaginary part. Also, it is difficult to improve the efficiency at approximately 13.9 nm with other materials, such as Si, Ni, W, and Pt. To confirm the efficiency enhancement effect by molybdenum overcoating on a gold-coated varied-line-spacing (VLS) holographic grating, the zeroth and first order diffraction efficiencies have been calculated at an angle of incidence of  $87^\circ$  at a wavelength of 13.9 nm, as shown in Fig. 1(a). The grating parameters are as follows: a 1/1200 mm grating constant, a 16 nm groove depth, and a 0.3 duty ratio [10]. Both the zeroth and first order diffraction efficiencies monotonically increase with the Mo thickness, however, once the thickness is in excess of 16 nm, they vary alternately with respect to the thickness; when one increases, the other decreases, and vice versa. Therefore, the Mo thickness has been determined to be 16 nm.



**FIGURE 1.** (a) Mo thickness dependence of diffraction efficiencies for the zeroth and first orders. (b) Comparison of measured diffraction efficiencies of the Au- and Mo-coated gratings at a grazing angle of incidence of  $3^\circ$  at a wavelength of 13.9 nm.

Two gold-coated VLS gratings were prepared, and a thin Mo layer was coated on one grating simultaneously along with a silicon wafer by means of ion beam sputtering. The Mo thickness on the silicon wafer was evaluated by curve fitting analysis of the X-ray reflectivity measured with the Cu- $K\alpha$  radiation, being 16.9 nm.

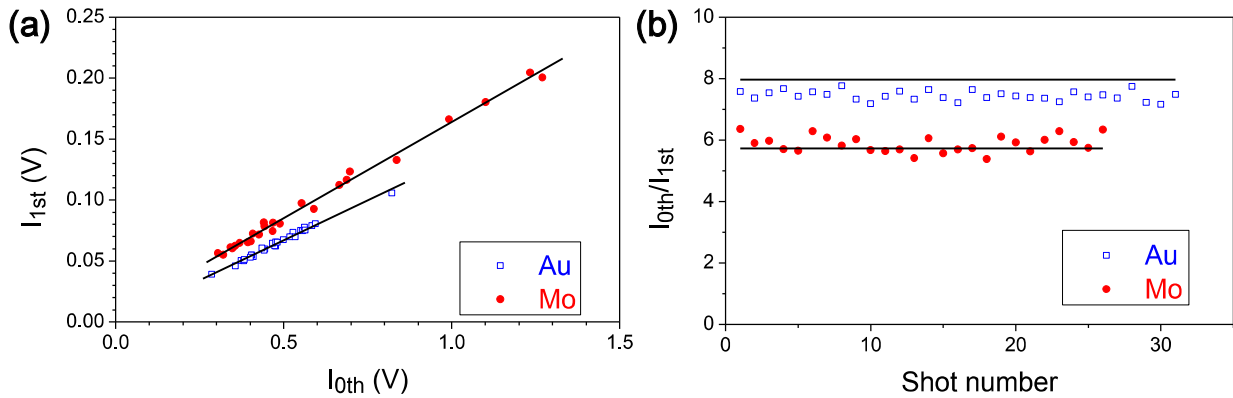
Absolute diffraction efficiency measurements were performed at the BL-11D of the Photon Factory, KEK, Japan. This beamline provides soft X-rays from 60 to 1200 eV and is equipped with a dedicated reflectometer. The beamline monochromator was set at a wavelength of 13.9 nm, and no bandpass filter was used for removing higher order diffracted light from the beamline. The beam size at the sample position in the reflectometer was  $60 \mu\text{m}$  (V)  $\times$  1 mm (H). The grating sample was set at a grazing angle of incidence of  $3^\circ$  which is the complementary angle, and the reflected and/or diffracted light from the grating was detected with an X-ray photodiode detector (AXUV100G Si/Zr, IRD Inc.) placed 280 mm away from the sample. The detector acceptance area of 10 mm-square was restricted to 5 mm (V)  $\times$  10 mm (H) to improve the angular resolution.

Figure 1(b) shows measured diffraction efficiencies of the Au- and Mo-coated gratings as a function of the detection angle. It is found that all the measured diffraction efficiencies of the 0th to 6th orders of the Mo grating are higher than those of the Au grating. Especially, the 0th order diffraction peak for use in downstream experiments reaches 74.6% from 69.5%, and the 1st one for beam intensity monitoring is increased to 13.0% from 8.7%. In addition, the Mo grating has decreased the baseline level, and therefore, the 0.5th order diffraction peak originating from the second order diffracted light of the beamline monochromator has been more clearly observed. Unfortunately, note that these two gratings are not identical, and there is the possibility that the absolute diffraction efficiencies are variable within individual samples.

## PERFORMANCE TEST OF GRATING-BASED BEAM INTENSITY MONITORS

Performance test of the gratings was conducted at an end station of the XRL beamline, QST, by using a new evaluation instrument that has the mount parameters compatible to the 1200 lines/mm holographic grating (Shimadzu, #30-002): the angle of incidence is  $87^\circ$ ; the distance from the source point to the grating center is 237 mm; the distance from the grating center to the image plane is 235 mm. The evaluation instrument consists of a grating sample, an entrance slit to set the angle of incidence, and two X-ray photodiodes (AXUV100G) for detecting the zeroth and first order diffracted light. The 13.9 nm XRL beam with a divergence of  $\sim 0.2$  mrad generated from the source point in double-target mode passes through a  $0.5\text{-}\mu\text{m}$  thick Zr filter to remove visible light and then is irradiated at an angle of incidence of  $\sim 3^\circ$  onto a spherical mirror (SM) having a 2-m radius of curvature placed at a distance of 2 m from the source point. The horizontally reflected light by the SM is introduced into the entrance slit of  $0.1\text{ mm (H)} \times 13.6\text{ mm (V)}$  in the evaluation instrument after being horizontally reflected by a plane mirror (M1) at  $\sim 45^\circ$ . The intensities of the zeroth and first order diffracted light are measured simultaneously with the respective photodiodes placed on the image plane.

As shown in Fig. 2(a), the Pearson's correlation coefficient has been measured between the intensities of the zeroth ( $I_{0\text{th}}$ ) and first ( $I_{1\text{st}}$ ) order diffracted light, resulting in 0.994 for the Au grating and 0.997 for the Mo grating. Figure 2(b) shows the intensity ratio ( $I_{0\text{th}}/I_{1\text{st}}$ ) plotted as a function of shot number. For the comparison, the results of absolute diffraction efficiency measurements are also represented by the solid lines (see Fig. 1(b)). Although there is a slight difference on account of angular misalignment of the Au grating, the intensity ratios are in good agreement with each other, regardless of light sources. These indicate that both the gratings work as beam intensity monitors for XRL.



**FIGURE 2** (a) Correlations between the intensities for the zeroth ( $I_{0\text{th}}$ ) and first ( $I_{1\text{st}}$ ) order diffracted lights of the Au- and Mo-coated gratings. (b) Plots of the intensity ratio ( $I_{0\text{th}}/I_{1\text{st}}$ ) versus shot number. The solid lines indicate the intensity ratios obtained by absolute diffraction efficiency measurements with synchrotron radiation, as shown in Fig. 1(b).

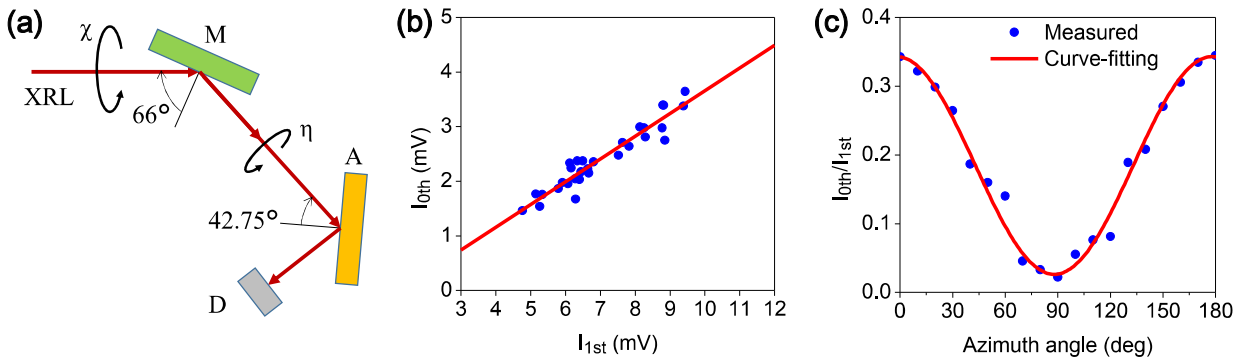
## ELLIPSOMETRY MEASUREMENTS

Ellipsometry measurements with the zeroth order diffracted light delivered from the Mo grating was carried out by using a newly developed rotating-analyzer ellipsometer capable of being equipped with a pre-mirror (M) and an analyzer mirror (A) made with Mo/Si multilayers, as shown in Fig. 3(a). The respective azimuth angles  $\chi$  and  $\eta$  of M and A can be moved in vacuum, but both the angles of incidence were fixed. The light introduced into the ellipsometer

was horizontally reflected by M at  $\chi = 0^\circ$  and then reached an X-ray photodiode (AXUV100G) after being reflected by the A. The performance of the multilayer mirrors M and A at angles of incidence of  $66.00^\circ$  and  $42.75^\circ$ , respectively, has been characterized with synchrotron radiation in advance [8].

As shown in Fig. 3(b), the correlation has been measured between the photodiode signals obtained with the ellipsometer ( $I_{0th}$ ) and the Mo grating-based beam intensity monitor ( $I_{1st}$ ) when A was fixed at  $\eta = 0^\circ$ , which corresponds to horizontal reflection because of  $\chi = 0^\circ$ . Although there is a slight fluctuation, a high correlation coefficient of 0.953 is obtained.

Figure 3(c) shows the intensity ratio  $I_{0th}/I_{1st}$  plotted as a function of  $\eta$  ranging from 0 to  $180^\circ$ . The S/N ratio is poor due to the asymmetric beam shape of the 0th diffracted light expanded in vertical direction, but the polarization state of the XRL beam was determined by curve fitting analysis, resulting in a linear polarization degree of 0.86 [8]. This is reasonable because the reflection by M1 at the incident angle close to the polarization angle of  $\sim 45^\circ$  mainly contributes to the improvement of the degree of linear polarization as opposed to the small polarization selectivity from SM and M in addition to the Mo grating.



**FIGURE 3.** (a) Schematic diagram of the rotating-analyzer ellipsometer: M, Mo/Si multilayer mirror; A, Mo/Si multilayer polarizer (analyzer);  $\chi$ , azimuth rotation of M;  $\eta$ , azimuth rotation of A; D, photodiode detector. (b) Correlation between the photodiode signals  $I_{0th}$  and  $I_{1st}$  measured at  $\chi = \eta = 0$ . (c) Intensity ratio  $I_{0th}/I_{1st}$  plotted as a function of  $\eta$  when  $\chi = 0$ .

## CONCLUSION

We have confirmed that a thin Mo layer overcoated on a gold-coated holographic grating improves the diffraction efficiency and works as a high-efficiency beam intensity monitor for 13.9 nm Ni-like Ag XRL. In addition, the practicality of the beam intensity monitor has been validated by determining quantitatively the polarization state of the XRL beam delivered from the Mo grating with rotating-analyzer ellipsometry.

## ACKNOWLEDGMENTS

Part of this work was conducted under the approval of the Photon Factory Program Advisory Committee (Proposal No.2014G531). This study was partially supported by Japan Society for the Promotion of Science KAKENHI Grant 23760040 and 15K04685.

## REFERENCES

1. H. Daido, *Rep. Prog. Phys.* **65**, 1513–1576 (2002).
2. M. Tanaka, M. Nishikino, T. Kawachi, N. Hasegawa, M. Kado, M. Kishimoto, K. Nagashima, and Y. Kato, *Opt. Lett.* **28**, 1680–1682 (2003).
3. A. Y. Faenov, N. A. Inogamov, V. V. Zhakhovskii, V. A. Khokhlov, K. Nishihara, Y. Kato, M. Tanaka, T. Pikuz, M. Kishimoto, M. Ishino, M. Nishikino, T. Nakamura, Y. Fukuda, S. V. Bulanov, and T. Kawachi, *Appl. Phys. Lett.* **94**, 231107 (2009).
4. T. Suemoto, K. Terakawa, Y. Ochi, T. Tomita, M. Yamamoto, N. Hasegawa, M. Deki, Y. Minami, and T. Kawachi, *Opt. Exp.* **13**, 14114–14122 (2010).

5. R. Sobierajski, R. A. Loch, R. W. E. van de Kruijs, E. Louis, G. von Blanckenhagen, E. M. Gullikson, F. Siewert, A. Wawro, and F. Bijkerk, *J. Synchr. Rad.* **20**, 249–257 (2013).
6. A. Depresseux, E. Oliva, J. Gautier, F. Tissandier, G. Lambert, B. Vodungbo, J-P. Goddet, A. Tafzi, J. Nejdil, M. Kozlova, G. Maynard, H. T. Kim, K. Ta Phuoc, A. Rouse, P. Zeitoun, and S. Sebban, *Phys. Rev. Lett.* **115**, 083901 (2015).
7. K. Tiedtke, J. Feldhaus, U. Hahn, U. Jastrow, T. Nunez, T. Tschentscher, S. V. Bobashev, A. A. Sorokin, J. B. Hastings, S. Möller, L. Cibik, A. Gottwald, A. Hoehl, U. Kroth, M. Krumrey, H. Schöppe, G. Ulm, and M. Richter, *J. Appl. Phys.* **103**, 094511 (2008).
8. T. Imazono, *Appl. Opt.* **56**, 5824–5829 (2017).
9. P. Karvinen, S. Rutishauser, A. Mozzanica, D. Greiffenberg, P.N. Juranić, A. Menzel, A. Lutman, J. Krzywinski, D.M. Fritz, H.T. Lemke, M. Cammarata, and C. David, *Opt. Lett.* **37**, 5073–5075 (2012).
10. T. Imazono, M. Koike, T. Nagano, H. Sasai, Y. Oue, S. Kuramoto, M. Terauchi, H. Takahashi, S. Notoya, T. Murano, E. M. Gullikson, “Experimental evaluation of a method to enhance the diffraction efficiency by overcoating diamond-like carbon (DLC) on soft X-ray laminar-type gratings,” in 12th International Conference on Synchrotron Radiation Instrumentation (SRI2015), AIP Conf. Proc. 1741, edited by Q. Shen et al. (American Institute of Physics, Melville, NY, 2016), pp. 040043-1–040043-4.
11. B. L. Henke, E. M. Gullikson, and J. C. Davis, “X-ray interactions: photoabsorption, scattering, transmission, and reflection at  $E = 50\text{--}30000$  eV,  $Z = 1\text{--}92$ ,” *At. Data Nucl. Data Tables* **54**, 181–342 (1993).