Gaudin, J.; Ozkan, C.; Chalupsky, J.; Bajt, S.; Burian, T.; Vysin, L.; CoppolaN.; Dastjani-Farahani, S.; Chapman, H.N.; Galasso, G.; Hajkova, V.; Harmand, M.; Juha, L.; Jurek, M.; Loch, R.A.; Moeller, S.; Nagasono, M.; Stoermer, M.; Sinn, H.; Saksl, K.; Sobierajski, R.; Schulz, J.; Sovak, P.; Toleikis, S.; Tiedtke, K.; Tschentscher, T.; Krzywinski, J. :

Investigating the interaction of x-ray free electron laser radiation with grating structure

In: Optics Letters (2012) OSA

DOI: 10.1364/OL.37.003033

Investigating the interaction of x-ray free electron laser radiation with grating structure

Jérôme Gaudin,[1,*] Cigdem Ozkan,[1] Jaromír Chalupský,[2] Saša Bajt,[3] Tomáš Burian,[2] Luděk Vyšín,[2] Nicola Coppola,[1] Shafagh Dastjani Farahani,[1] Henry N. Chapman,[3] Germano Galasso,[1] Věra Hájková,[2] Marion Harmand,[4] Libor Juha,[2] Marek Jurek,[5] Rolf A. Loch,[6] Stefan Möller,[7] Mitsuru Nagasono,[8] Michael Störmer,[9] Harald Sinn,[1] Karel Saskl,[10] Ryszard Sobierajski,[5] Joachim Schulz,[1,3] Pavol Sovak,[11] Sven Toleikis,[4] Kai Tiedtke,[4] Thomas Tschentscher,[1] Jacek Krzywinski[7]

[1]European XFEL GmbH, Albert-Einstein-Ring 19, Hamburg, D-22671, Germany

[2]Institute of Physics, Academy of Sciences of the Czech Republic, Na Slovance 2, Prague 8, 182 21, Czech Republic

[3]Center for Free-Electron Laser Science, DESY, Notkestr. 85 Hamburg, D-22607, Germany

[4] HASYLAB/DESY, Notkestr. 85 Hamburg, D-22607, Germany

[5]Institute of Physics, Polish Academy of Sciences, Al. Lotników 32/46, Warsaw, PL-02-668, Poland

[6]SLAC National Accelerator Laboratory, 2575 Sand Hill Road, Menlo Park, California 94025, USA

[7]Dutch Institute for Fundamental Energy Research, PO Box 1207, 3430 BE Nieuwegein, The Netherlands

[8]RIKEN/SPring-8 Kouto 1-1-1, Sayo, Hyogo, 679-5148 Japan

[9] Helmholtz Zentrum Geesthacht, Institute of Materials Research, Max-Planck Str. 1, D-21502 Geesthacht, Germany

[10]Institute of Materials Research, Slovak Academy of Sciences, 040 01 Kosice, Slovak Republic

[11]Institut of Physics, P. J. Šafárik University, Park Angelinum, 04154Kosice, Slovak Republic

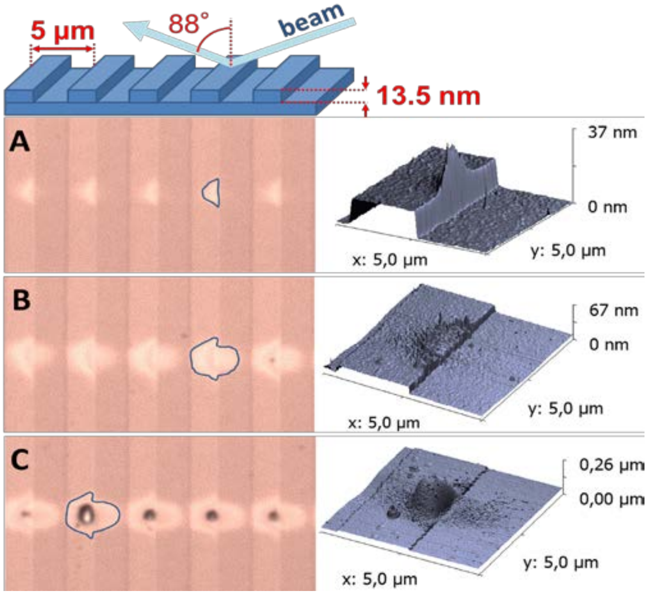
**Corresponding author: jerome.gaudin@xfel.eu*

Received Month X, XXXX; revised Month X, XXXX; accepted Month X, XXXX; posted Month X, XXXX (Doc. ID XXXXX); published Month X, XXXX

The interaction of free electron laser pulses with grating structure is investigated using 4.6 ± 0.1 nm radiation at the FLASH facility in Hamburg. For fluences above 63.7 ± 8.7 mJ / cm², the interaction triggers a damage process starting at the edge of the grating structure as evidenced by optical and atomic force microscopy. Simulations based on solution of the Helmholtz equation demonstrate an enhancement of the electric field intensity distribution at the edge of the grating structure. A procedure is finally deduced to evaluate damage threshold.

OCIS Codes: 000.0000, 999.9999

X-ray free electrons lasers (XFELs) have demonstrated to be powerful tools for new, high impact research in different scientific fields. Motivated by successful experiments, new facilities (European XFEL, FLASH 2, LCLS 2) with improved properties in terms of energy per pulse over an extended wavelength range, are currently under construction. Certain schemes promise to deliver up to tens of milli-Joule pulses. Soft x-ray spectroscopy experiments require monochromatized light which can be provided through a grating-based monochromator. State-of-the-art grating is currently limited to length around 250 mm resulting in quite high fluence impinging on the grating's surface. Damage of the grating is major concern for the design of the beamline. Investigating and understanding damage mechanism of grating structures is thus of a fundamental importance for the development of the next generation XFELs. Predicting the damage threshold is difficult in case of periodically structured surfaces, like gratings, as the electric field intensity distribution is highly non-homogeneous at the surface and can lead to enhanced damage mechanism as evidenced in



the optical domain [1]. In this contribution, we report on both experimental and theoretical investigations of XFEL radiation interaction with grating structure.

Fig. 1 Top: Schematic of the interaction of the experiment. Below: DIC microscopy (left) and AFM (right) measurements for three different fluences 356 (A), 806 (B) and 1115 mJ/cm² (C)

The experiment was performed at the FLASH facility in Hamburg [2]. The sample was placed, under vacuum, in the focus of a 2 m focal length mirror. The pulse duration was in the 80-150 fs range, and the radiation wavelength was measured to be 4.6 ± 0.1 nm. This error bar corresponds to the uncertainty on the absolute value of the wavelength and not to the pulse to pulse jittering, which has also be measured and found to be negligible (~1e-3 nm). The beam was impinging the sample at the grazing angle $\alpha = 2° \pm 0.1$, following a procedure described in [3]. The 200 l/mm (5 µm periods) grating sample was produced by ion etching of a 1 mm thick Si wafer, with a duty ratio of 0.4. The groove depth was measured to be 13.5 nm. These parameters reproduce a real grating currently used in the SXR beamline at the LCLS [4]. The etched wafer was then coated with 45 nm of amorphous carbon (a-C), which is a typical coating for XFEL optics. Atomic force microscopy (AFM) measurement confirms that the coating exactly reproduces the ion etched profile. The grating sample, as well as a mirror-like flat sample also made of 45 nm thick a-C coated on Si substrate, was exposed to single pulses with varying pulse energy. For each pulse, the pulse energy was measured with a gas detector monitor. The pulse energy monitor was located upstream all optical components, e.g. the beamline transmission should be evaluated so the real pulse energy impinging on the sample can be known. As already stated the radiation is wavelength was known with an accuracy of ± 0.1 nm. In this wavelength domain the beamline transmission is evaluated to range from 0.20 (at 4.50 nm) to 0.46 (at 4.70 nm). This large variation is primarily due to the two up-stream carbon coated mirrors present in the beamline which are very sensitive around the carbon K-edge (at 4.37 nm). In this analysis we assume therefore a wavelength of 4.60 nm corresponding to a beamline transmission of 0.39.

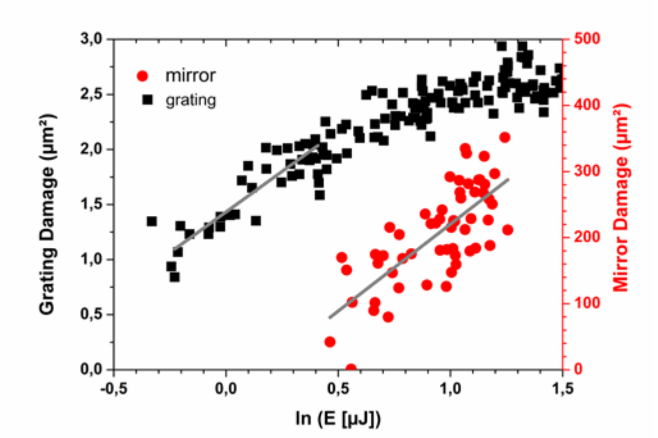


Fig. 2. Damaged area on flat mirror-like sample (red dots, right vertical scale) and grating (black square, left vertical scale)

The exposed samples were analyzed ex-situ by optical differential interference contrast (DIC) microscopy which is sensitive to variations of optical refractive index, hence to any phase change, evidenced by a change of color. For each irradiated sample regions, the area showing a color change (encircled areas shown in Fig. 1) in the microscope image was measured. For both flat and grating sample, the energy damage threshold (E_{th}) was determined by plotting the damaged area versus pulse energy, as shown in Fig. 2. A line fit through the point gives a threshold for grating $E^{G}_{th} = 0.40 \pm 0.04$ µJ and $E^{M}_{th} = 1.17 \pm 0.16$ µJ for the flat sample. The error bars corresponds to the confidence on the fit, at the 4.60 nm wavelength. We then calculate the ratio $E^{M}_{th} / E^{G}_{th} = 2.92 \pm 0.69$, which will be used for comparison with the model later, and is independent of the beamline transmission value.

The damage fluence threshold (F_{th}) is retrieved from the values of E_{th} and by making the following assumption. The beam profile was carefully characterized using imprints in PMMA sample and following the procedure described in [5]. The effective area is found to be $A_{eff} = 22 \pm 2$ µm². We assumed that beam footprint in case of grazing incidence is equal to the projected area, e.g. $A_{eff} / \sin(2)$. We obtained: $F^{G}_{th} = 63.7 \pm 8.7$ mJ / cm² for the grating and $F^{M}_{th} = 186.6 \pm 29.9$ mJ / cm² for the flat sample.

Fig. 1A, obtained with DIC microscopy, shows the onset of the damage on the edge of the grating structure. The AFM measurements confirm this observation, and show the extension of the damage first on the top of the groove as the fluence is increasing. The damage in the bottom part of the grating structure happens at higher fluence as can be seen in Fig. 1B. At the highest fluence values (see figure 1 C), black dots become visible due to the complete removal of the a-C coating and melting of the Si substrate.

To gain deeper insight in the understanding of the beam / grating interaction, an accurate model has to be used. As stated in the introduction, the electric field distribution at the surface of a grating can be highly non-homogeneous, especially while dealing with a coherent laser beam. We simulated the deposited energy distribution in the grating by solving the Helmholtz equation in a paraxial approximation. Assuming that the refractive index of the medium is nearly equal to 1 (which is true for the photon energy considered here) then the propagation of the scalar field ψ can be expressed as:

$$\frac{\partial \Psi(\hat{r},\hat{z})}{\partial \hat{z}} = \frac{i}{2} \cdot \frac{\partial^2 \Psi(\hat{r},\hat{z})}{\partial \hat{r}^2} + \delta\varepsilon(\hat{r},\hat{z}) \cdot \Psi(\hat{r},\hat{z}) \qquad (1)$$

$$\hat{r} = rk \,,\, r^2 = x^2 + y^2 \,,\, \hat{z} = zk \,,\, k = 2 \cdot \pi / \lambda$$

λ is the wavelength and $\delta(r,z)$ describes the difference between the dielectric constant of vacuum and the medium. The mathematical form of the Eq. 1 is identical to the time dependent, 2D Schrödinger equation. Many effective methods exist to solve this type of equation; we chose a version of beam propagation method which applies a split operator technique [6,7]. The grating is modelled by considering a 45 nm thick layer of a-C on Si-substrate. The grating profile used in the simulations as a boundary is the real profile measured with AFM. As a result the model also takes into account possible effect of the micro-roughness. The real and imaginary part of $\delta(r,z)$ were taken from the CXRO database [8]. We used a Gaussian beam profile as the initial condition. The incident angle and the photon energy were the same as in the experiment. The evolution of $|\psi(r,z)|^2$ is shown in Fig. 3. A standing wave builds up close to the grating's surface. The inset in Fig. 3 depicts the distribution of the absorbed energy in the grating's structure. The maximum absorption is taking place at the edge of the grating structure where the onset of damage is experimentally observed. Interestingly, micro-roughness does not increase the maximum of absorbed energy by more than few percent as it was confirmed by simulations on a smooth surface. Moreover, similar simulations (not shown) were also performed with a mirror-like flat surface. The ratio of the maximum energy absorbed in the grating to the absorbed energy in the flat mirror is found to be $\gamma = 3.37$, γ can be directly related to the ratio of the damage thresholds. Both values agree, within the error bar of the experiment, demonstrating the quantitative accuracy of our approach. One should underline that γ does not reflect an enhancement of the intrinsic absorption process, but the non-homogeneous field distribution at the surface of a grating leading to local enhancement of the absorbed energy.

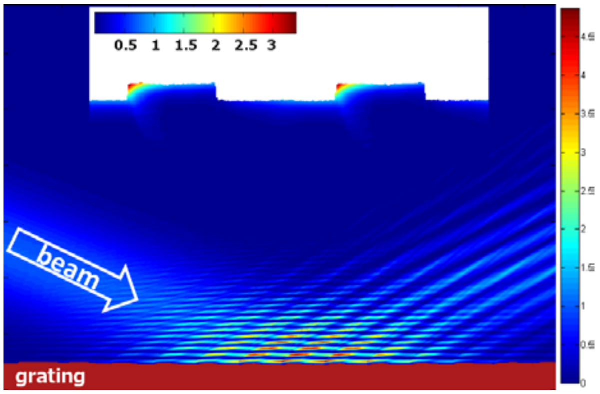


Fig. 3 X-ray intensity distribution $|\psi(r,z)|^2$ close to the grating surface. The beam comes from the left. Inset: energy distribution absorbed in the grating. Both colour scales are in arbitrary units.

In conclusion, we have investigated the interaction of 4.60 nm XFEL radiation with grating structure. An accurate description based on Helmholtz equation shows that the specific field distribution at the surface leads to an enhancement of the absorbed energy at the edge of the laminar grating structure. The model provides a good qualitative and quantitative description of the experimental results. From a practical point of view, when designing a soft x-ray monochromator for an XFEL beamline, it is crucial to consider the fluence damage threshold values. We propose to first extrapolate $F^{M}_{th}(\alpha)$ for a flat mirror-like surface using published data (for example Ref. [3]). Next, simulations should be performed taking into account the exact geometry of the grating, to obtain γ. Finally the damage threshold on grating is deduced from the relation $F^{G}_{th}(\alpha) = F^{M}_{th}(\alpha) / \gamma$.

This work was financially supported within projects P108/11/1312, LA08024, CZ.1.05/1.1.00/483/02.0061, Z.1.07/2.3.00/483/20.0087, P205/11/0571, P208/10/2302, and ME10046.

References

1. S. Hocquet, J. Neauport, and N. Bonod, Appl. Phys. Lett. **99**, 061101 (2011).
2. W. Ackermann et al. Nat. Photonics **1**, 336 (2007)
3. J. Chalupský, V. Hájková, V. Altapova, T. Burian, A. J. Gleeson, L. Juha, M. Jurek, H. Sinn, M. Störmer, R. Sobierajski, K. Tiedtke, S. Toleikis, Th. Tschentscher, L. Vyšín, H. Wabnitz, and J. Gaudin, Appl. Phys. Lett. **95**, 031111 (2009)
4. P. Heimann, O. Krupin, W. F. Schlotter, J. Turner, J. Krzywinski, F. Sorgenfrei, M. Messerschmidt, D. Bernstein, J. Chalupský, V. Hájková, S. Hau-Riege, M. Holmes, L. Juha, N. Kelez, J. Lüning, D. Nordlund, M. F. Perea, A. Scherz, R. Soufli, W. Wurth, and M. Rowen, Rev. Sci. Instrum. **82**, 093104 (2011)
5. J. Chalupský, J. Krzywinski, L. Juha, V. Hájková, J. Cihelka, T. Burian, L. Vyšín, J. Gaudin, A. Gleeson, M. Jurek, A. R. Khorsand, D. Klinger, H. Wabnitz, R. Sobierajski, M. Störmer, K. Tiedtke, and S. Toleikis, Opt. Express **18**, 27836 (2010)
6. O. K. Ersoy, *Diffraction, Fourier Optics and Imaging* (John Wiley and Sons, 2007)
7. H. J. W. M. Hoekstra, Opt. Quantum Electron. **29**, 157 (1997)
8. B.L. Henke, E.M. Gullikson, and J.C. Davis, Atomic Data and Nuclear Data Tables **54**, 181 (1993)

Full references for referees.

[1] S. Hocquet, J. Neauport, and N. Bonod, "The role of electric field polarization of the incident laser beam in the short pulse damage mechanism of pulse compression grating," Appl. Phys. Lett. **99**, 061101 (2011)

[2] W. Ackermann, G. Asova, V. Ayvazyan, A. Azima, N. Baboi, J. Bähr, V. Balandin, B. Beutner, A. Brandt, A. Bolzmann, R. Brinkmann, O. I. Brovko, M. Castellano, P. Castro, L. Catani, E. Chiadroni, S. Choroba, A. Cianchi, T. Costello, D. Cubaynes, J. Dardis, W. Decking, H. Delsim-Hashemi, A. Delserieys, G. Di Pirro, M. Dohlus, S. Düsterer, A. Eckhardt, T. Edwards, B. Faatz, J. Feldhaus, K. Flöttmann, J. Frisch, L. Fröhlich, T. Garvey, U. Gensch, Ch. Gerth, M. Görler, N. Golubeva, H.-J. Grabosch, M. Grecki, O. Grimm, K. Hacker, U. Hahn, J. H. Han, K. Honkavaara, T. Hott, M. Hüning, Y. Ivanisenko, E. Jaeschke, W. Jalmuzna, T. Jezynski, R. Kammering, V. Katalev, K. Kavanagh, E. T. Kennedy, S. Khodyachykh, K. Klose, V. Kocharyan, M. Körfer, M. Kollewe, W. Koprek, S. Korepanov, D. Kostin, M. Krassilnikov, G. Kube, M. Kuhlmann, C. L. S. Lewis, L. Lilje, T. Limberg, D. Lipka, F. Löhl, H. Luna, M. Luong, M. Martins, M. Meyer, P. Michelato, V. Miltchev, W. D. Möller, L. Monaco, W. F. O. Müller, O. Napieralski, O. Napoly, P. Nicolosi, D. Nölle, T. Nuñez, A. Oppelt, C. Pagani, R. Paparella, N. Pchalek, J. Pedregosa-Gutierrez, B. Petersen, B. Petrosyan, G. Petrosyan, L. Petrosyan, J. Pflüger, E. Plönjes, L. Poletto, K. Pozniak, E. Prat, D. Proch, P. Pucyk, P. Radcliffe, H. Redlin, K. Rehlich, M. Richter, M. Roehrs, J. Roensch, R. Romaniuk, M. Ross, J. Rossbach, V. Rybnikov, M. Sachwitz, E. L. Saldin, W. Sandner, H. Schlarb, B. Schmidt, M. Schmitz, P. Schmüser, R. Schneider, A. Schneidmiller, S. Schnepp, S. Schreiber, M. Seidel, D. Sertore, V. Shabunov, C. Simon, S. Simrock, E. Sombrowski, A. Sorokin, P. Spanknebel, R. Spesyvtsev, L. Staykov, B. Steffen, F. Stephan, F. Stulle, H. Thom, K. Tiedtke, M. Tischer, S. Toleikis, R. Treusch, D. Trines, I. Tsakov, E. Vogel, T. Weiland, H. Weise, M. Wellhöfer, M. Wendt, I. Will, A. Winter, K. Wittenburg, W. Wurth, P. Yeates, V. Yurkov, I. Zagorodnov, and K. Zapfe, "Operation of a freeelectron laser from the extreme ultraviolet to the water window," Nat. Photonics **1**, 336 (2007).

[3] J. Chalupský, V. Hájková, V. Altapova, T. Burian, A. J. Gleeson, L. Juha, M. Jurek, H. Sinn, M. Störmer, R. Sobierajski, K. Tiedtke, S. Toleikis, Th. Tschentscher, L. Vyšín, H. Wabnitz, and J. Gaudin, "Damage of amorphous carbon induced by soft x-ray femtosecond pulses above and below the critical angle" Appl. Phys. Lett. **95**, 031111 (2009)

[4] P. Heimann, O. Krupin, W. F. Schlotter, J. Turner, J. Krzywinski, F. Sorgenfrei, M. Messerschmidt, D. Bernstein, J. Chalupský, V. Hájková, S. Hau-Riege, M. Holmes, L. Juha, N. Kelez, J. Lüning, D. Nordlund, M. F. Perea, A. Scherz, R. Soufli, W. Wurth, and M. Rowen "Linac Coherent Light Source soft x-ray materials science instrument optical design and monochromator commissioning" Rev. Sci. Instrum. **82**, 093104 (2011)

[5] J. Chalupský, J. Krzywinski, L. Juha, V. Hájková, J. Cihelka, T. Burian, L. Vyšín, J. Gaudin, A. Gleeson, M. Jurek, A. R. Khorsand, D. Klinger, H. Wabnitz, R. Sobierajski, M. Störmer, K. Tiedtke, and S. Toleikis "Spot size characterization of focused non-Gaussian X-ray laser beams" Opt. Express **18**, 27836 (2010)

[6] O.K. Ersoy, *Diffraction, Fourier Optics and Imaging* (John Wiley and Sons, 2007)

[7] H. J. W. M. Hoekstra, "On beam propagation methods for modeling in integrated optics", Opt. Quantum Electron., **29**, 157 (1997)

[8] B.L. Henke, E.M. Gullikson, and J.C. Davis. "X-ray interactions: photoabsorption, scattering, transmission, and reflection at E=50-30000 eV, Z=1-92", Atomic Data and Nuclear Data Tables Vol. **54** (no.2), 181-342 (July 1993)