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Investigation of damage induced by intense femtosecond XUV pulses in silicon crystals by means of white beam synchrotron section topography

W. Wierzchowski [a,*], K. Wieteska [b], D. Klinger [c], R. Sobierajski [c], J.B. Pelka [c], D. Żymierska [c], T. Balcer [a], J. Chalupský [d], J. Gaudin [e], V. Hájková [d], T. Burian [d], A.J. Gleeson [f], L. Juha [d], H. Sinn [e], D. Sobota [g], K. Tiedtke [h], S. Toleikis [h], T. Tschentscher [h], L. Vyšín [d], H. Wabnitz [h], C. Paulmann [h]

[a] *Institute of Electronic Materials Technology, Wólczyńska 133, Warsaw 01-919, Poland*
[b] *National Centre for Nuclear Research, Soltana 7, Otwock-Świerk 05-400, Poland*
[c] *Polish Academy of Sciences, Institute of Physics, al. Lotników 32/46, Warsaw 02-668, Poland*
[d] *Institute of Physics, Academy of Sciences of the Czech Republic, Na Slovance 2, 182 21, Prague 8, Czech Republic*
[e] *European XFEL, DESY, Notkestr., 85 D-22607 Hamburg, Germany*
[f] *CCRLC Daresbury Laboratory, Warrington, Cheshire WA4 4AD, United Kingdom*
[g] *Jan Kochanowski University, Institute of Physics, ul. Świętokrzyska 15, Kielce 25–406, Poland*
[h] *HASYLAB/DESY, Notkestr., 85 D-22607 Hamburg, Germany*

HIGHLIGHTS

- The vicinity of ablation craters induced by XUV FEL radiation was studied.
- Disturbed crystal lattice was found below the ablation craters.
- The evaluated depth extension was in the range of 30–100 μm.
- Approximation of droplet-like inclusion was used for numerical simulations.
- Melting and recrystallization may be responsible for observed defects.

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ABSTRACT

Silicon crystalline samples were exposed to intense single pulses of XUV radiation ($\lambda = 13.5$ nm) what lead to melting and ablation of the surface material. The deformation field around craters along the whole thickness of silicon wafers was observed by means of the synchrotron transmission section topography using the beam perpendicular to the surface of the sample. The geometrical shape and depth extension around craters was evaluated based on numerous, dense series of section topographs spaced by 10 μm. In the topographs we observed the direct image connected with the boundary of the crater associated with some deformation of the Kato fringes. The evaluated depth extension, different for individual craters, was in the range of 30–100 μm. The depth values were confirmed also by evaluations based on the Bragg case section topographs.

It was possible to reproduce the contrast of the craters in transmission section topographs by numerical simulation based on integration of the Takagi–Taupin equations. The damage of the crystal defects connected with craters was approximated as droplet-like inclusions.

1. Introduction

The important question associated with the use of new 4th generation of short wavelength radiation sources, the Free Electron Lasers (FELs), refers to the interaction of the extremely intense beams generated by these devices with solids. It is expected, that the irradiation with very intense femtosecond pulses can create very strongly excited states. Their characteristic feature is a highly reduced influence of optical nonlinearities at the frequencies above the plasma frequencies (Hau-Riege et al., 2007; Krzywinski et al., 2007). The experiment included generation of the damages by means of irradiation with beam coming from the

* Corresponding author.
E-mail address: wierzc_w@itme.edu.pl (W. Wierzchowski).

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Free-electron LASer in Hamburg (FLASH) operating at 13.5 nm wavelength and focussed by an ellipsoidal mirror onto the surface of crystalline silicon. The damage obtained with XUV–FEL irradiation at monocrystaline silicon wafer and some other materials was previously studied with the micro-beam (Pelka et al., 2004) and nano-beam (Pelka et al., 2009) X-ray diffraction methods together with AFM and Nomarski contrast microscopy. The first study made it possible to gauge the range of damage, that was estimated to spread around the ablation craters, together with accompanying strain field, no more than 120–150 μm, while the second experiment provided direct observation of structural changes, mainly due to recrystallization of the material.

In our previous experiment exploiting the X-ray topography, the use of back reflection section and projection topography enabled us to reveal many important features of the strain fields connected with the craters (Wierzchowski et al., 2011). It was in particular possible to demonstrate a significant similarity of the observed strain field to that of rod-like inclusion. The last results suggested unexpectedly large depth extension of the strain field connected with the craters.

In the present paper we report on the studies of the craters generated by XUV pulses by means of the synchrotron transmission section white beam topographs. The use of the transmission section topography seemed be very promising, as it can provide the information about the strain field connected with the craters from the whole thickness of the sample. Also the interpretation of the results visible in so called “direct” contrast (Authier, 1967) seems to be relatively simple and univocal. The results from the transmission section topography are also compared with those obtained by means of back reflection section topography.

The completion of the section topographs were the simulation of contrast obtained using more adequate approximation of crater by droplet-like inclusion. The approximation was chosen since the interaction of the ion or electron beam with the solid matter cause “droplet like” distribution of the induced defects.

2. Experimental

The experiments were performed at the free electron laser in Hamburg (FLASH) in Germany. It delivers pulses of approx. 50 fs duration. For all experiments, a procedure similar to the one described in details in Ref. (Sobierajski et al., 2013) was followed. The optically polished monocrystaline Si wafers with (111) surface orientation were placed within a high vacuum experimental chamber in the focus of the ellipsoidal mirror and exposed to single XUV pulses at normal incidence. The wavelength of the incident radiation was 13.5 nm. The energy of each pulse was varying due to stochastical properties of the radiation generation process (Ayvazyan et al., 2003; Saldin et al., 2000). It was measured by means of gas ionization detector. For the purpose of further investigation a number of irradiation spots was chosen. In this set the minimum fluence of approx. 1 J/cm^2 was a few times higher than the threshold fluence of the surface modifications, while the maximum fluence was approx. 5 J/cm^2.

The most important synchrotron topographic method used for determination of depth extension of the craters was transmission section topography realized with a very narrow (5 μm) and 7 mm horizontally extended white beam perpendicular to the surface of the sample. The experiments were performed at F1 station of the DORIS III synchrotron at HASYLAB using the beam from the bending magnet with critical energy E_c=16.6 keV and high energy range better than 31 keV. The scheme of the experiment is shown in Fig. 1. The depth evaluation is possible due to the fact that a characteristic dark direct contrast is formed in the vicinity of the incoming beam intersecting the crystal.

Experiments were performed at the numerous dense series of section topographs spaced by 10 μm provided a precise scan enabling the evaluation of geometrical shape and depth extension of some various craters. The accuracy of the depth evaluation depends mainly on the measurement of the crystal thickness which corresponds to the width of all section images.

Another possibility of depth evaluation offers the Bragg-case section topography as it is illustrated in Fig. 2. Also in this case the contrast of the craters is direct; hence the topographs reproduce the “droplets” intersected by narrow incident beam. When the craters are regularly spaced with the same distance each from another the number of the reproduced craters is dependent on the depth extension. If the number of the reproduced craters is n and their distance is s then the depth extension $D=nstg\alpha$, where α is

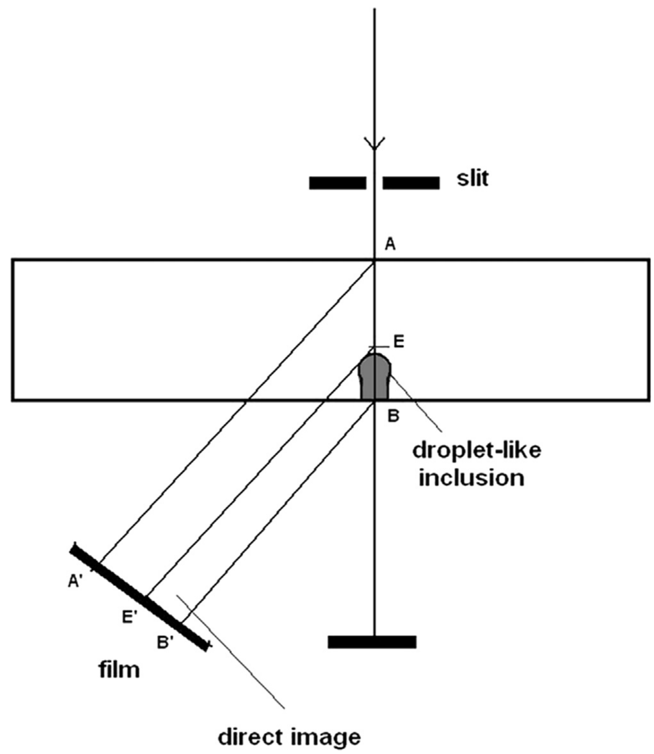

Fig. 1. Schema of the direct image formation in transmission section diffraction topography. A and B denote the points where incident beam perpendicular to the surface respectively enters and exit the sample while the E denotes the point corresponding to the maximal depth extension of the craters. The letters with primes denote respectively the images of the mentioned points recorded at the film.

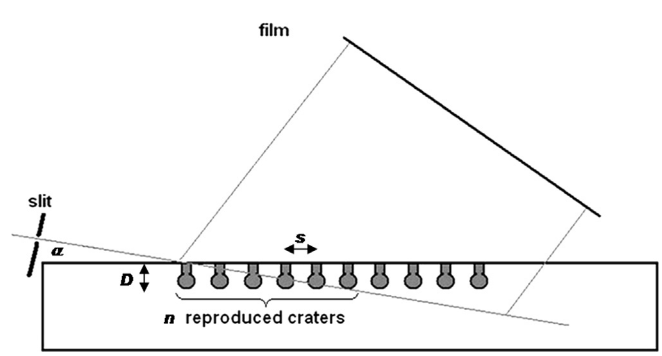

Fig. 2. Schema illustrating the formation of the crater images and their visibility in Bragg-case section topography. n—number of the reproduced craters, s—distance between craters, D—depth extension and α—the glancing angle at which the beam enters the sample (presently equal to 4°).