ESRF	**Experiment title:** **Structure modifications in materials irradiated by ultra-short pulses of VUV free electron laser**	**Experiment number:** **HS 2658**
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Report:

The objective of the project was to characterize structure modifications induced on sample surfaces by irradiation with free electron laser (FEL) working in the XUV region. The samples – Si(001) wafers, deposited on sapphire (110) Au films and amorphous SiO_2 – were irradiated at XUV FLASH, the FEL facility at DESY, Hamburg, with the quantum energy centered around 38.15 eV (λ=32.5 nm), in single short pulses of only 25 fs and of peak power up to 1 GW. The FEL beam was focused on the sample surfaces to microspots of size 10-30 μm. The energy density in the spots varied from 0.05 J/cm^2 up to 2.5 J/cm^2, the upper value being far above the ablation threshold for the materials.

The structure was investigated by x-ray diffraction. The intense x-ray microbeam at ID-13 beamline of only ca. ~250 nm diameter, of the wavelength λ = 0.97626 Å and divergence of 1 mrad was applied. 2D diffraction patterns were recorded in transmission mode with the 2D MARCCD-165 detector equipped with 2048 x 2048 matrix of pixels, while the sample was step-scanned by the beam with step-size of 200-1000 nm along chosen paths throughout craters irradiated with single laser pulses of various fluencies.

Post-irradiation surface morphology has been examined by a number of techniques, including the optical microscopy with Nomarski contrast and AFM pattern record (Nanoscope III), related to fluency data of the irradiation and compared with microdffraction results.

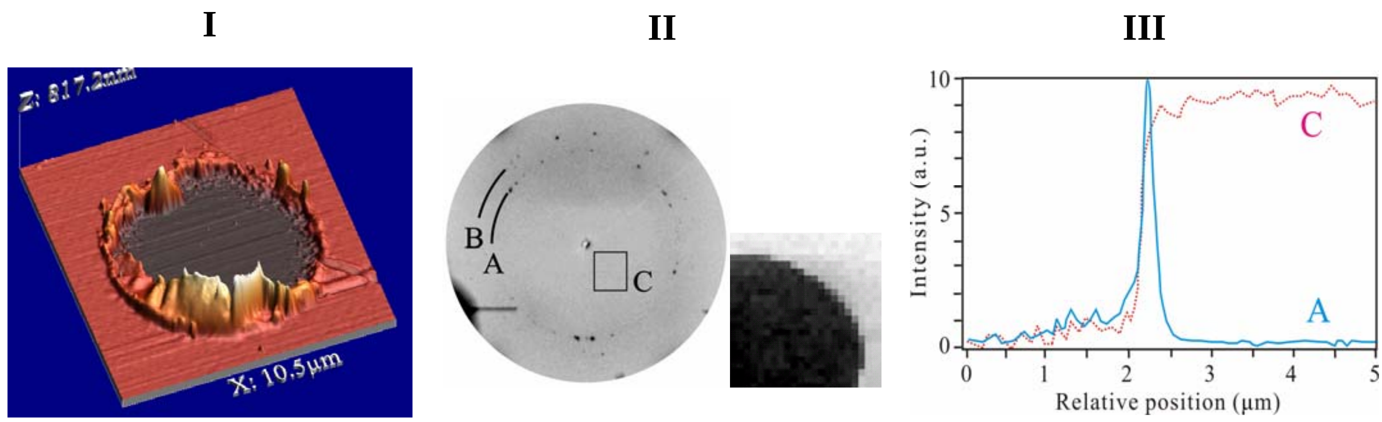

Fig. 1. I - AFM pattern of a single-shot crater damaged with the irradiation fluency of 0.28 J/cm^2 XUV radiation from FLASH in the Au film 90 nm thick deposited on sapphire. **II -** 2D diffraction pattern recorded exactly in the embankment at the edge of crater, A and B are rings of gold 111 and 200 reflections, respectively. The rectangle C defines the background intensity integration area. Background intensity distribution map reflects the thickness variations of the gold film (dark area inside ablation crater shown in the right part of the picture). **III -** Integrated intensity of gold reflection (111) (A) and integrated background intensity (C) recorded along a straight path starting in the center of the crater shown in (I).

An example of x-ray microdiffraction and AFM results is shown in Fig. 1**.** It has been found that in the ablated places on gold film surface a step-like, complete removal of gold film occurs, reflected both in a relevant background intensity drop inside the craters and in a lack of gold peaks in diffraction signal (see Fig 1.III). The polycrystalline phase occurs only in narrow strip areas close to the crater boundaries ~200-300 nm wide, set upright as walls (embankments) up to few micrometers high and outlining the crater boundaries, as it was found by comparison with AFM patterns. The size of gold crystallites in these embankments was found, from diffraction line profiles analysis, to be not smaller than 15 nm. In some places the profiles showed additional fraction of gold crystallites of sizes below 2-3 nm. Small shifts observed in the peak positions can suggest changes in the lattice constant of the gold crystallites. In the centers of craters irradiated with the highest fluencies a signal from the sapphire polycrystalline phase, as well as from small gold residues was found. The observed morphology and structural properties suggest that the gold crystallites at the crater edges are remains of a native crystalline structure of gold film rather than a newly crystallized material from gold melted by irradiation.

Similar narrow crystalline embankments were found around ablation craters on the silicon surface. Also in this case, residues of crystalline material were found in the central parts of craters irradiated with higher fluencies. Contrary to this, no traces of any diffraction peaks in the areas of irradiation damage on amorphous SiO_2 surface were found.

The obtained results support the ablation models assuming that damage induced in solids with ultrafast, intense XUV pulse occurs on very rapid time-scales with material beneath the crater surfaces structurally almost unaffected of damage.