Applications of Synchrotron Radiation Scattering to Studies of Plasma Facing Components at Siberian Synchrotron and Terahertz Radiation Centre

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Abstract. The paper presents an overview of plasma-material interaction studies at the Siberian Synchrotron and Terahertz Radiation Centre. The measurements of recrystallization and surface texturing are demonstrated. The growth of grain size after the exposure by electron beam was detected using two-dimensional diffractometry. The orientation of crystal structure was detected in the tungsten irradiated by deuterium plasma. The residual stresses in irradiated tungsten were measured. The predicted structure of deformations after pulsed heat load is confirmed. First in-situ experiments confirmed the presence of crystal plane rotation effect during a pulsed heat load.

INTRODUCTION

Intensive flows of plasma particles and radiation on the first wall and divertor plates are expected in the next generation of the experimental fusion reactors [1]. A number of plasma-material interaction (PMI) phenomena influence the crystal structure of material and consequently can be measured using synchrotron radiation (SR) scattering diagnostics. The results of the measurements of the grain size, the orientation of crystal structure and residual deformations are discussed. The parameters are important for the several PMI issues (mechanical failure, heat removal, tritium retention, etc.). The paper presents an overview of PMI studies at the Siberian Synchrotron and Terahertz Radiation Centre [2, 3].

EX-SITU EXPERIMENTS

The X-ray diffraction on tungsten samples exposed to plasma and electron beams was measured at the stations "Diffractometry in hard X-rays" and "Precision diffractometry and anomalous scattering" of the SSTRC. The ex-situ

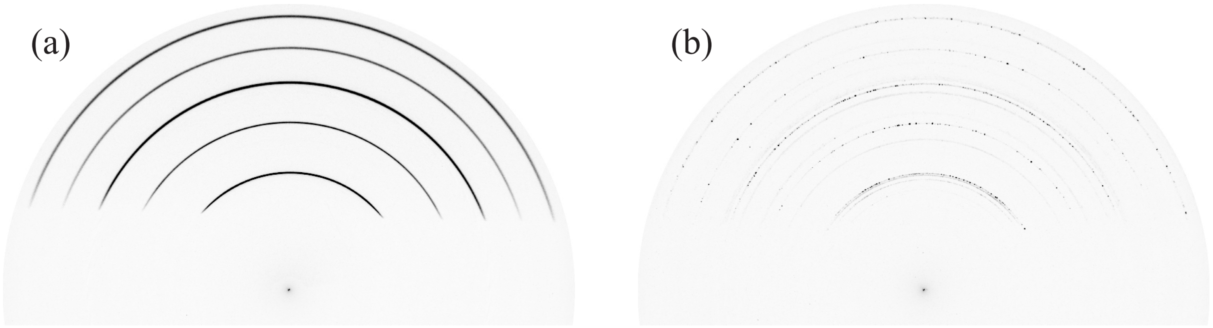

FIGURE 1. Two-dimensional diffractograms of tungsten composite. (a) initial composite; (b) composite irradiated by electron beam (electron energy of 31 keV, power density of 1.6 GW m^{-2}, duration of 260 μs, 1000 pulses).

experiments yielded results on recrystallization, surface texturing and residual stress.

Recrystallization

One of the major issues in the degradation of divertor plates is the recrystallization of tungsten during repetitive heat loads [4]. The surface layer of the plates becomes more brittle due to the recrystallization and hence the cracking threshold decreases. It is well known that the growth of grains can be controlled in composite materials [5]. That is why we intend to study recrystallization of composites based on tungsten. As first step, we examined the modification of tungsten sample under a multiple-pulse electron gun impact. As we are going to study composite materials further, the sample was fabricated using the hot pressing technique. Tungsten powder with a mean grain size of 10 μm was pressed at a temperature of 1350°C and a pressure of 50 MPa during 60 minutes. The two-dimensional reflection diffractograms of the composite were measured before and after irradiation by the electron beam (electron energy of 31 keV, power density of 1.6 GW m^{-2}, duration of 260 μs, 1000 pulses). The breaking of tungsten diffraction rings into separate dots after pulsed heat loads corresponds to a significant increase in the average size of the tungsten grains (Fig. 1). So, the result of recrystallization was detected. A quantitative analysis of the results for calculation of the average grain size is in progress.

Surface texturing

A diffractometry analysis of initial tungsten and that exposed to deuterium plasma at the LENTA facility was carried out [6]. The conditions of the last sample exposure follow:

- Ion energy: 250 eV,
- Exposure duration: $1.3 \cdot 10^4$ s,
- Fluence: $1.95 \cdot 10^{25}$ m^{-2},
- Thickness of sputtered layer: 1.6 μm,
- Sample temperature: lower than 100°C.

The variation in the ratio of the measured intensity of different diffraction peaks was more than tenfold (Fig. 2). Such a significant effect evidently indicates the appearance of crystal structure orientation in the surface layer. The columnar structure of solidified tungsten melt may cause such degree of surface texturing [7]. However, the tungsten was not heated appreciably. The surface texturing caused by the interaction of the tungsten with the hydrogen plasma may be important for several issues of plasma material interaction due to the change in the surface layer properties. In particular, the certain orientation of the crystal structure may causes significant change of sputtering yield and diffusion of hydrogen and helium in the material. Besides that, the heat removal and thresholds of mechanical failure should be revised due to the anisotropy of thermal conductivity and mechanical strength of material with oriented crystal structure.

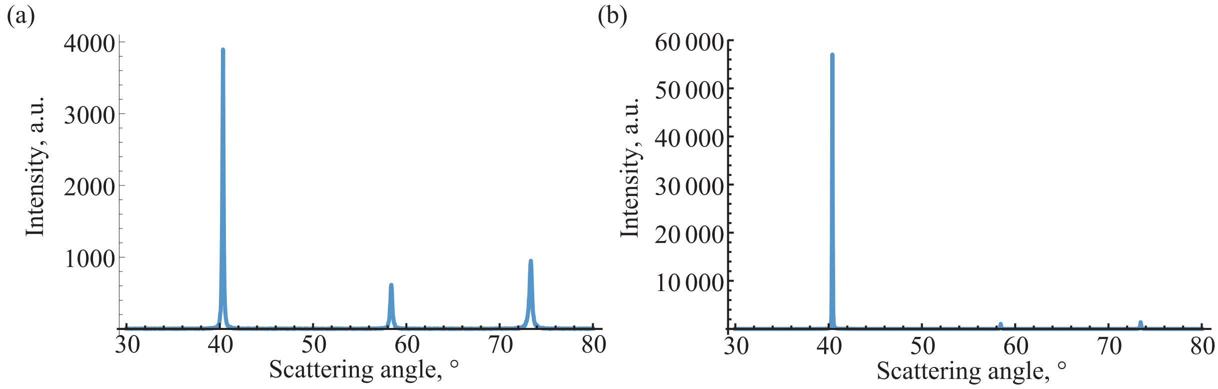

FIGURE 2. Theta-2theta scans of tungsten (photon energy of 8.048keV). (a) initial tungsten; (b) tunsgten irradiated by deuterium plasma at room temperature [6].

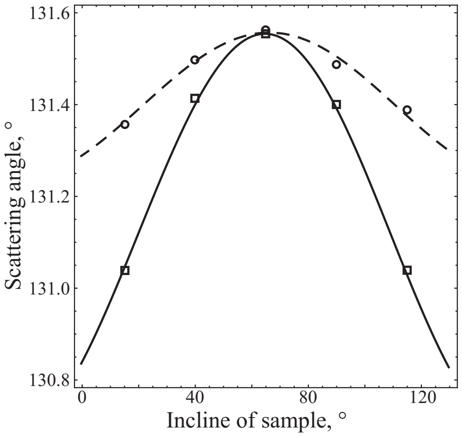

FIGURE 3. The measured data and the approximation of the scattering angle dependance on the sample inclination. The squares and the solid line correspond to $\phi = 0°$; the circles and the dashed line correspond to $\phi = 90°$.

Residual stress

Cracking after pulsed heat load is typical to tungsten [8]. The reason for the crack formation is the residual tensile stress caused by plastic deformation during a pulsed heat load [9]. The residual deformations in the surface layer of tungsten sample irradiated at GOL-3 near [10]. The heat load to the sample was close to threshold of cracking to provide residual stress close to ultimate tensile strength. The deformation was measured using the variation of scattering angle 2θ on inclination of sample relative to incident SR beam and rotation around the rotation around normal of surface:

$$\frac{1}{\sin\theta} = \frac{2d_0}{n\lambda}(1 - \varepsilon_{xx}\sin^2(\psi-\theta)\cos^2\phi - \varepsilon_{yy}\sin^2(\psi-\theta)\sin^2\phi - \varepsilon_{zz}\cos^2(\psi-\theta) -$$

$$2\varepsilon_{xy}\sin^2(\psi-\theta)\cos\phi\sin\phi - 2\varepsilon_{xz}\sin(\psi-\theta)\cos(\psi-\theta)\cos\phi - 2\varepsilon_{yz}\sin(\psi-\theta)\cos(\psi-\theta)\sin\phi), \qquad (1)$$

where d_0 is the interplanar distance, n is the interference order, λ is the wavelength, ε_{ij} is the deformation tensor, ψ is the angle of the sample inclination and ϕ is angle of the sample rotation around its surface normal. The components of the deformation tensor and the interplanar distance were varied to fit the measured data (Fig.3). The absence of the perpendicular to surface stress was used to eliminate the ambiguity:

$$\varepsilon_{zz} = -\frac{\sigma}{1-\sigma}\left(\varepsilon_{xx} + \varepsilon_{yy}\right), \qquad (2)$$

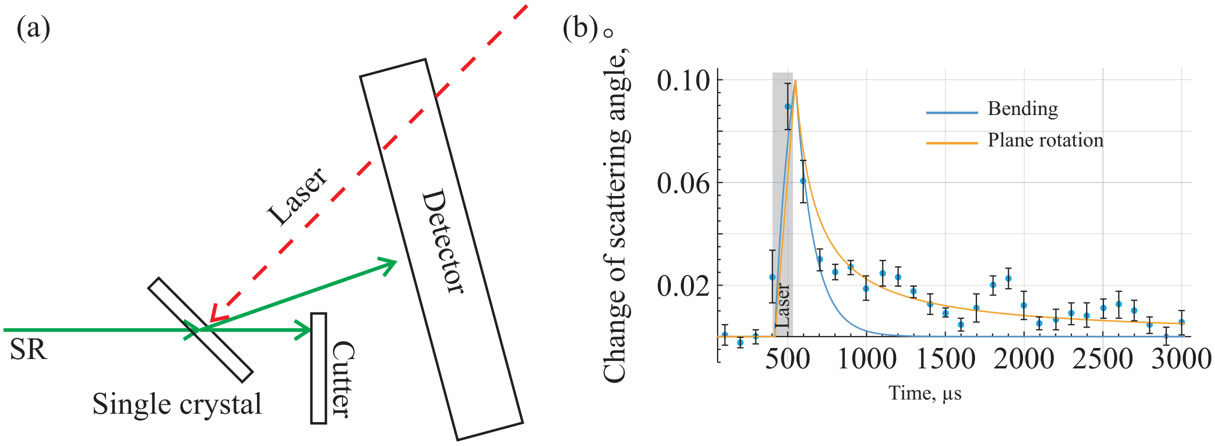

FIGURE 4. (a) The scheme of the experiment; (b) The time dependencies of the change in the scattering angle. The errors are the confidence intervals for the average value calculated on the basis of several measurements. The grey background marks the time interval of the laser heating. The curves correspond to different theoretical models.

where σ is the Poisson's ratio. The residual stresses were calculated using the measured deformations. The obtained value was close to the ultimate tensile stress [10]. The interplanar distance obtained by the fitting differs from the nonperturbed value is about 0.014%. The measurements confirm the predicted structure of residual deformations and stresses [9].

IN-SITU EXPERIMENTS

Experimental scheme

Diagnostics on the base of SR scattering have a number of advantages:

- Dynamical measurements during pulsed processes,
- Measurements inside the material,
- Spatial resolution inside the material.

The first in-situ PMI experiments on the VEPP-4 SR source are aimed at dynamical measurements of tungsten deformations during pulsed heat load. The basic variant of the experimental scheme realizing all the listed advantages of SR scattering diagnostics is drawn in Fig. 4(a). The Bragg geometry is a back-up option. It does not allow one to achieve a spatial resolution and requires a polished single crystal surface, but in this case the thickness of the sample is not limited by the SR attenuation.

The main expected effect is the rotation of the crystal due to anisotropic deformation. The rotation angle depends on the local temperature [9].

Experimental setup

The pulsed heating is simulated by the Nd:YAG laser. The 1 J laser beam is focused into an about 1mm spot and heats the tungsten surface up to about 1000°C. The 100 J laser is under construction.

The sample orientation is set by two high precision motorized goniometric cradles. The goniometers allow one to change the angle between the crystal plane and the initial SR beam and turn the diffraction peak to the plane of the one-dimensional detector DIMEX. The development of the silicon detector for increasing the sensitivity is in progress. The list of the experiment parameters follows:

- SR cross-section: 0.5 mm × 0.5 mm.
- Laser diameter: 1 mm.
- Laser beam energy: 1 J.

- Laser duration: 140 μs.
- Sample: (111) single crystal tungsten.
- Diffraction plane: (110).
- Sample thickness: 250 μm.
- Distance to detector: 300 mm.

Results

In order to confirm the existence of the crystal plane rotation effect, we measured the decay of the change in the scattering angle. The decay of the bending effect is much faster than the plane rotation one. The bending is proportional to the moment of the force, which decays exponentially with a typical time of the equalizing of the temperature across the sample (about 200 μs). The effect of crystal plane rotation decays according to the power law with an exponent of -1. The comparison of the experimental results with the fittings corresponding to the bending and plane rotation models demonstrates that the effect of the crystal plane rotation is at least comparable with the bending effect during the pulsed heat load (Fig. 4 (b)).

SUMMARY

Ex-situ experiments on the SR scattering for PMI studies yielded the following: detection of recrystallization after pulsed heat loads, detection of surface texturing after exposure to deuterium plasma and measurement of residual stresses. The growth of the average grain size was detected at the exposed to electron beam tungsten. The experiment is preliminary one in the study of the reduction of recrystallization by the using of two-component composite.

The orientation of the crystal structure was found in the surface layer of tungsten irradiated by deuterium plasma. The effect may be important for several issues of plasma material interaction due to the change in the surface layer properties.

The measurements of residual deformations after simulation of pulsed heat loads were carried out as preliminary experiment of the dynamic measurements of deformations and stresses during pulsed heat load. The measurements confirmed the predicted structure of residual deformations and stresses. The first in-situ experiments demonstrated the change of the diffraction scattering angle during pulsed heat load. The measurement of the change decay confirmed the presence of the crystal plane rotation effect.

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