STRUCTURE MODIFICATION OF DIFFERENT GRAPHITE AND GLASSY CARBON SURFACES UNDER HIGH POWER ACTION BY HYDROGEN PLASMA

A. A. Shoshin[1,2], A.V. Arzhannikov[1,2], A.V. Burdakov[1], K.N. Kuklin[1], I.A. Ivanov[1,2], K.I. Mekler[1], S.V. Polosatkin[1,2], V.V. Postupaev[1,2], K.S. Raspopin[3], A.F. Rovenskikh[1], P.A. Simonov[3], S.L. Sinitsky[1,2], V.N. Snytnikov[3]

[1]*Budker Institute of Nuclear Physics, 11 Lavrentjev Avenue, Novosibirsk, 630090, Russia*
[2]*Novosibirsk State University, 2 Pirogova Street, Novosibirsk, 630090, Russia*
[3]*Boreskov Institute of Catalysis, 5 Lavrentjev Avenue, Novosibirsk, 630090, Russia*
e-mail: A.A.Shoshin@inp.nsk.su

The studies of exposing of targets by hot-electron plasma exhaust from the multi-mirror trap GOL-3 are carried out. The results of Raman and SEM studies of surface modification of carbon materials with different ordering range of sp2 fraction (different graphite and glassy carbon) exposed by pulsed hot hydrogen plasma with energy density up to 8 MJ/m^2 are presented. It is shown that on the depth up to ~80 nm due to this exposing there are obtained two effects – micro structural destruction of the surface and extension of crystallite size of sp2 phase, i.e. ordering of nanostructure.

I. INTRODUCTION

Carbon based engineering materials are used for different areas of science and technology, in particularly for plasma dumping. High power density loads engage to pick out well-ordered graphite composites possessed by high heat conductivity (CFCs and other). However they characteristics must be stable under long action by stream of high-energy particles. Such studies with different carbon materials are carried out for a long time (see e.g. [1,2]). According to referenced data a nanostructural modification of the materials under high-power action is observed. At the same time this process can lead to both disordering and extension of crystallite areas. The analysis of the studies shows that surface amorphization is observed due to different defects accumulation at relatively low heat input - under ~0.1 MJ/m^2. In a case of higher heat input the ordering of crystallite structure takes place, one possible mechanism of them is thermal graphitization [3].

II. EXPERIMENT

The studies of plasma confinement in multi-mirror trap GOL-3 placed in the Budker Institute of Nuclear Physics [4] and its heating by powerful relativistic electron beam (energy 0.8 MeV, energy content up 200 kJ, duration 8 μs) are carried out. Plasma temperature after heating stage reaches ~2 keV. In the exit of the plasma machine the stream of hot plasma and passed electron beam is dumped by collector, placed in splay magnetic field [5]. Common energy density is ~2 MJ/m^2 corresponds to ELMs type I in ITER. The total energy density reached the target was measured by heat radiation obtaining from its surface and by bolometer [5].

In that conditions the different carbon materials, large grained (electrode) graphite, fine grained graphite's MPG-6 and CGD, glassy carbon SU-2000 were exposed. Structure of glassy carbon is amorphous and MPG-6 can be characterized as nanostructured polycrystal. For studying the carbon specimens were made in the form of plates of typical size ~20 mm and thickness 2-3mm.

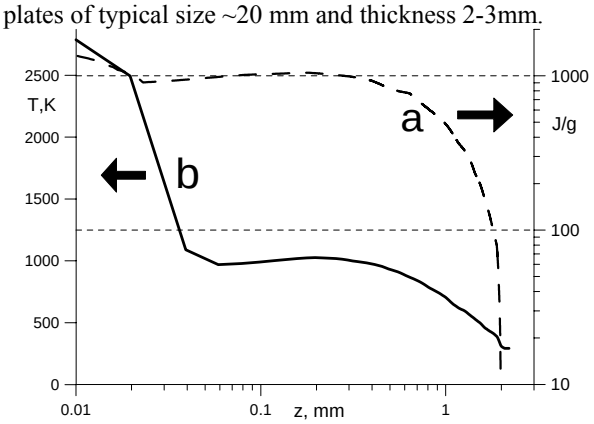

Fig. 1. Energy release (dashed) and temperature (solid) distribution versus carbon target depth after REB ending.

Calculated energy release distribution versus carbon target depth is shown in Fig. 1a. One can see, that high-energy electrons (0.1-1 MeV) with ranges ~1 mm heat bulk of target up to ~1000 K. Superficial layer on the depth of ~20 μm (see Fig. 1b) overheats by energy release from epithermal electrons and hot plasma ions. The value of volumetric energy release in this area can be greater

than enthalpy of phase change of microstructural graphite destruction (~10 kJ/g, [6]). However simulation of heat transport by DISWALL code [6], shows that maximal temperature does not exceed 3000 K that is below sublimation temperature (4100 K).

III. RESULTS

For obtaining of nanostructure modification the Raman spectroscopy was used. The method based on measuring of profiles of G- and D- vibration graphite modes. Changes of they intensities and widths by Tuinstra-Koenig correlation [7, 8] give the opportunity to estimate crystallite size of sp2 phase (L_a):

$$C(\lambda)/L_a(Å) = I_D/I_G,$$

where: $\lambda = 514.5$ nm – wave length of excited light, $C(\lambda) \approx 44$Å. The method gives averaged L_a size on the carbon layer depth of 50-100 nm depending on material.

Before exposing graphite specimens MPG-6 were burnished for micro structural modification obtaining (see SEM photo Fig. 3a). Its initial Raman spectrum is shown in Fig. 2 (top curve). One can see sharp G- (1623 cm^{-1}) and D- (1360 cm^{-1}) peaks are present. So additional D' peak is obtained at 1623 cm^{-1}. Spectrum is fitted by asymmetric Breit-Vigner-Fano profile (G) and two Lorenzian profiles (D, D') with 28 and 50 cm^{-1} widths correspondingly. The spectrum allows to characterize this material in accordance with [2] as nano-structured graphite. Peaks ratio is $I_D/I_G = 0.81$ that allow to estimate [1] graphite crystallite size of 50 Å.

The specimen was exposed by four repeated plasma impacts up to 8 MJ/m^2 integral heat load. The exposed surface has defects of erosion of ~10 μm characteristic dimension obtained by SEM (see Fig. 3b). It agrees with MPG-6 grains size covering the range of 10-100 μm. This process can be obtained because of grains binding energy some less than microstructural graphite destruction ~10 kJ/g. In Raman spectrum changes take place after irradiation. Width G- and D-peak diminished to 5-8 cm^{-1}, asymmetry of G peak reduced and peaks ratio decreased up to 0.17 that testifies arising of crystallite size up to 260 Å.

The Raman spectrum of glassy carbon is shown in Fig. 4 (bottom). In the spectrum G- (1594 cm^{-1}) and D- (1360 cm^{-1}) peaks with widths of 95 и 90 cm^{-1} correspondingly are seen. Obtained spectrum allows us to characterize the studied material in accordance with [7] as carbon with amorphous structure. For such materials the Tuinstra-Koenig correlation stops to hold. And for estimation of graphite (sp2-phase) ordering sizes the correlation proposed in [7] can be used. The result is about 9.6 Å.

The specimen was exposed by three repeated plasma impacts up to 6 MJ/m^2 total heat load. In Fig. 5 the surface SEM photos before (a) and after (b) exposing are shown. Some scales ~100 nm thick are seen on surface after exposing. In Raman spectrum of glassy carbon changes are seen after exposing (Fig. 4, top curve). D peak shifted to 1335 cm^{-1}, widths both peaks diminished to 80 cm^{-1} and peaks ratio increased to 0.72. That testifies to graphitization of specimen surface and allows estimating sp2-phase ordering sizes as 11.5 Å.

For large grained graphite a crystallite size of 160 Å before irradiation and 210 Å after plasma exposed at the same condition were calculated by Raman spectra. These are for graphite CGD this is 180 and 200 Å respectively. So quality of initial graphite influences on following modification.

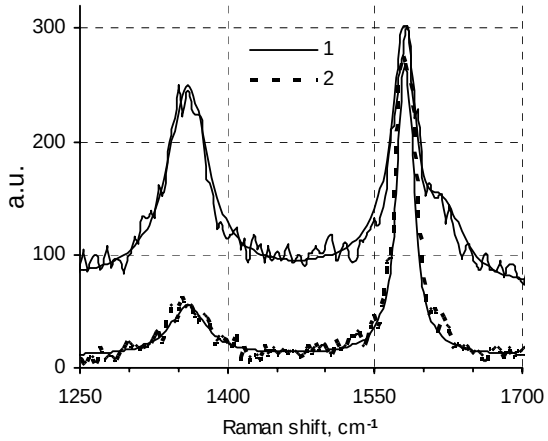

Fig. 2. Fitted Raman dispersion spectra of fine grained graphite MPG-6 before (solid line) and after (dashed) exposing (8 MJ/m^2 total heat load) and the corresponding fits.

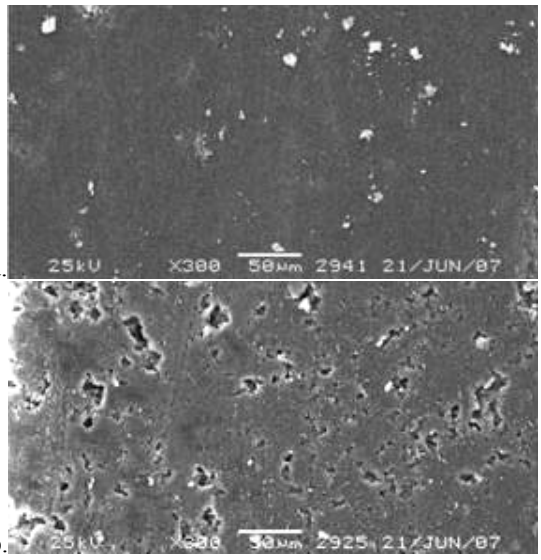

Fig. 3. SEM image of fine grained graphite MPG-6 before (a) and after (b) exposing.

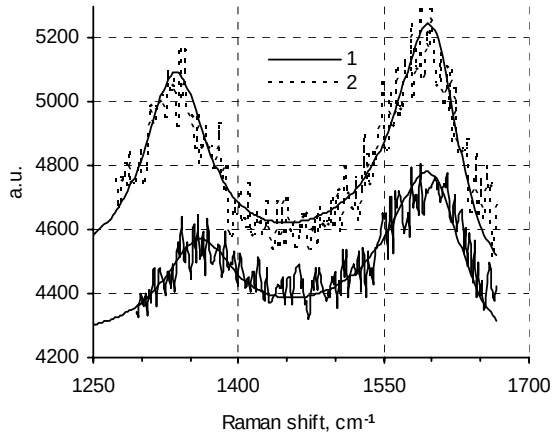

Fig. 4. Fitted Raman dispersion spectra of glassy carbon before (solid line) and after (dashed) exposing.

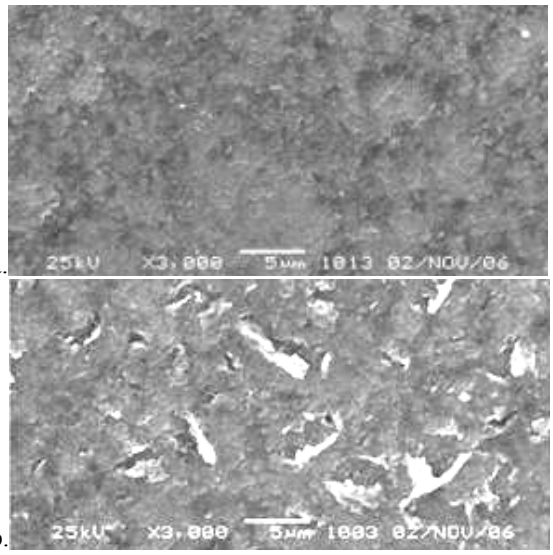

Fig. 5. SEM image of glassy carbon before (a) and after (b) exposing (6 MJ/m^2 total heat load).

IV. DISCUSSION

In [3] it is shown that carbon materials can be divided into graphitized and un-graphitized by heat treatment at 2000-3000 K. The typical graphitized materials have texture with turbostatic orientation reached over sizeable distances. Under heating it assists to gradual azimuth rotation of some layers up to matching with neighbors organizing in scope the graphite structure. In the typical un-graphitized materials the local well-ordered zones are appreciably smaller in size and placed confusedly therefore ordering increase under heat treatment results in small crystallite arising with mutual chaotic ordering.

The obtained results show the change of superficial structure modification of carbon materials under hot-electron plasma exposing of 6-8 MJ/m^2 density load for several milliseconds. In both graphites and glassy carbon specimens the extension of sp2-phase crystallites size is obtained by Raman spectroscopy. But because of glassy carbon belongs to un-graphitized materials the graphitization value is rather small.

Mechanism responsible for structure ordering can be thermal graphitization. However in [3] was pointed that this process has a long characteristic time. In particular for enlargement of L_a up to 100 nm for initially turbostatic orientation one needs a day at 2700 K and some minutes at 3300 K. As was mentioned above the calculated surface temperature of targets reaches ~3000 K. But structure modification processes near surface of target can lead simultaneously to reduction of heat transport between grains and increase of surface temperature. That can accelerate the graphitization process as well.

V. CONCLUSIONS

The studies of behavior of essential structurally-different carbon materials, fine-grained graphite and glassy carbon, under hot-electron plasma exposing were carried out. The integral heat load was 6-8 MJ/m^2 at impact time of ~5 milliseconds. The Raman spectra from targets surface shown graphitization process of both substances on the depth at least of ~50 nanometers. It appears by enlargement of sp2-phase crystallites size.

The most probable mechanisms of specimens superficial graphitization are thermal treatment and re-deposition of carbon from dense gas-dust phase being close to the surface, caused by target destruction by powerful plasma stream.

ACKNOWLEDGMENTS

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