# Study of Radiation-Damaged Fusion Materials under High-Power Plasma Stream

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Abstract. Plasma-facing materials (PFM's) of a fusion reactor will be affected by high heat flux, fast particles and 14 MeV-neutron irradiation. All these factors are crucial for the lifetime of the reactor components. This paper is devoted to the experimental studying the radiation damage effect on erosion of materials under plasma impact. To obtain a high level of radiation damage, we simulated an accumulation of radiation damage under fast neutron irradiation by fast ions with energies in the interval of 1-60 MeV accelerated on the cyclotron at Kurchatov Institute. Using this method we can accumulate the radiation damage at the level equivalent to fast neutron effect at the dose of up to  $10^{22}$  neutron/cm<sup>2</sup> in a few days operation of the cyclotron. Both carbon materials and tungsten were taken for the study as the targets: MPG-8 (Russian graphite), SEP NB-31 (ITER PFM candidate) and W (99,95% wt). Irradiation on the cyclotron has been performed by 5 MeV carbon ions for carbon materials and 3-4 MeV alpha particles for tungsten. The plasma experiments have been performed on the materials having accumulated 0,1-10 dpa of radiation damage. Plasma erosion was studied on the linear plasma simulator LENTA. Irradiated samples were exposed to a steady-state deuterium plasma at 100 eV ( $D^+$  ions) on carbon materials and 250 eV on tungsten to dose of up to  $10^{21}$ - $10^{22}$  ion/cm<sup>2</sup>. Surface microstructure modification has been observed and comparison was made of damaged and non-irradiated materials. The evidences of radiation damage influence on the erosion process have been found by analysis of deformation, surface modification and erosion data. The study of erosion characteristics in plasma showed enhancement of erosion yield on carbon materials and structure damage on tungsten. New experimental approach developed in this work to explore the plasma-facing materials for accounting of neutron effect and the results obtained appear to be important for the further studies of the combined plasma and neutron irradiation effect on fusion PFM's .

#### 1. Introduction

Plasma-facing materials (PFMs) of a fusion reactor will be exposed to high heat flux, fast particle emission from plasma and 14 MeV-neutron irradiation. All these factors are crucial for the lifetime of the reactor first wall and divertor, which is mainly limited by erosion of PFMs. Important data on the plasma erosion have been collected for non-irradiated materials to date and extensive experimental basis has been formed for predictions of the lifetime of the plasma facing components. However, no experimental data exists that would make possible taking into account neutron factor in those predictions. At the same time the fast neutrons produce serious radiation damage in materials during long operation of a fusion reactor and they may also play an important role as additional limiting factor for the PFM lifetime. Estimations of radiation damage for power fusion reactor fall in the range from a few tens to hundred dpa. Therefore experimental investigation of plasma impact on the materials at high level of radiation damage is extremely demanded.

This paper describes a new experimental approach to the study of plasma erosion aiming to account for the radiation damage effect and presents the first experimental results on the surface erosion of high-level radiation-damaged materials. (The start of this activity was first announced in our previous paper [1]). Preparation of material samples with high level of accumulated radiation damage is a difficult experimental task [2]. There are different principal approaches to obtain high-level radiation damage in materials. The first one relates to a fusion neutron source of a sufficiently high intensity, but it is not yet realized today.

Second, fission neutrons from a fast reactor may be taken. In this case a long irradiation time about one-year-scale period is needed to accumulate radiation damage at a high enough level. Finally, fast charged particles from accelerators (protons as well as heavy ions) suit well for experimental modeling of radiation damage produced in fusion materials. The latter method has been taken in these investigations and it was developed on the basis of experimental facilities at Kurchatov Institute.

### 2. Experiment and results

### 2.1. Method, facilities and materials.

Integrated experiments have been performed to study plasma erosion effect of radiationdamaged materials. The first stage of the experiments consisted in the production of high level of radiation damage in test materials. At the second stage these irradiated materials were subjected to plasma exposure. Radiation damage resulting from neutron irradiation effect was simulated by fast ions accelerated on the cyclotron at Kurchatov Institute (similar to the method used for SiC in [3]). The cyclotron provides acceleration of different ion species H<sup>+</sup>, He<sup>+</sup>, Li<sup>+</sup>,C<sup>+</sup> etc. in the energy interval 1-60 MeV. By this method we can produce a high level of radiation damage and obtain in a few days operation of the cyclotron the effect equivalent to fast neutron irradiation at the dose of up to 10<sup>22</sup> neutron/cm<sup>2</sup>. The accumulation of radiation damage takes place in this case in a surface layer of several microns depending on the type of ions, their energy and irradiated material.

Linear plasma machine LENTA [1] was used at the second stage of these experiments to process the irradiated materials in deuterium plasma and to study their surface erosion in condition relevant to SOL of a tokamak reactor. The plasma simulator LENTA generates plasmas in a steady state discharge powered by electron beam thus providing the ion current on the surface of materials under study at  $j_{ion}=10^{17}-10^{18}$  ion/cm<sup>2</sup>s (N<sub>e</sub>=10<sup>12</sup>-10<sup>13</sup> cm<sup>-3</sup>, T<sub>e</sub>=1-20 eV). Energy of the plasma ions was controlled by bias potential at the levels relevant to divertor conditions. Plasma exposures were performed step-by-step in order to reach in each of them deuterium ion fluence on the surface about  $10^{21}$  ion/cm<sup>2</sup>. Erosion effect was measured by weight loss method and analysis of the sample surface microstructure was made with SEM after each plasma run.

We have started these experiments on carbon materials because their application is still considered in ITER. The CFC candidate SEP NB-31 was taken along with Russian fine grain graphite MPG-8 and pyrographite quasi-single crystal. Then tungsten was also included as proposed for the ITER and as supposed for the next step reactors. It was W (99.95% wt) close by composition to the ITER candidate grade.

### 2.2. Carbon materials

**Irradiation by high-energy carbon ions.** Carbon ions  ${}^{12}C^+$  accelerated on the cyclotron to 5 MeV were taken to produce radiation damage in samples of three grades of graphites: SEP NB-31, MPG-8 and pyrographite [4]. The high level of radiation damage obtained in the materials was due to the choice of the ion species and to the high ion fluence received by the samples during irradiation of several days. By this, three levels of radiation damage 1 dpa, 5 dpa and 10 dpa in average over the damaged layer were obtained in the samples of each carbon material. The  ${}^{12}C^+$  ion doses necessary to reach these values were  $10^{17}$  ion/cm<sup>2</sup>,  $5 \cdot 10^{17}$  ion/cm<sup>2</sup> and  $10^{18}$  ion/cm<sup>2</sup> correspondingly. Distribution of the accumulated defects in the

surface layer is given in Fig. 1 where calculated curve of primary radiation defects produced in graphites by 5 MeV  ${}^{12}C^+$  ions as a function of depth is shown (SRIM program [5]).



Fig. 1. Calculated profile of the primary radiation defects D (dpa) as a function of depth for a carbon material with density  $\rho = 1.7 \text{ g/cm}^3$  after irradiation with 5 MeV carbon ions to the dose  $5 \cdot 10^{17}$ ion/cm<sup>2</sup>( $D_{max} = 65$  dpa lies at ~ 5  $\mu$ m,  $\langle D \rangle = 9,7$  dpa).

Sputtering effect and radiation-induced deformation (swelling) took place on the materials along with the radiation damage produced by the ions. Modification of surface structure of the SEP NB-31 sample is shown in Fig. 2 where the boundary between irradiated and non-irradiated areas is presented for the 10 dpa sample. The development of the surface is seen on the irradiated part (left).

Considerable linear deformation of the irradiated graphites has been detected by profilometer. The MPG-8 was found as the most radiation resistant in our case while the composite SEP NB-31 has shown large values of deformation:  $\Delta H = 20 \ \mu m$  on 10 dpa sample ( $10^{18} \ cm^{-2}$ ) (compare with 5  $\mu m$  ion range).



*Fig.2.* Border of irradiated area on SEP NB-31 surface (10 dpa): damage zone is to the left (scale 100  $\mu m$ ).

**Plasma experiments.** The graphite samples irradiated on the cyclotron were then exposed to steady-state deuterium plasma on the LENTA plasma simulator. The exposure parameters were taken as follows: deuterium ion current density about 10 mA/cm<sup>2</sup>, ion energy 100 eV (negative bias), sample temperature during plasma operation  $\leq 40$  C. The carbon materials were processed in the plasma in two steps 1 hour each. The layer of about a half fast ion penetration depth was eroded during the first step (about 2-3 µm), and the layer of the maximal radiation damage was eroded in the second plasma exposure (~3-7 µm) [6]. Weight losses were measured after plasma runs and surface microstructure was analyzed.

Changes were found both after each plasma exposure. Example of the SEP NB-31 surface irradiated to 10 dpa after plasma bombardment is shown in Fig. 3b.



Fig. 3. Surface of the SEP NB-31 sample: a) in initial state and b) after irradiation to 10 dpa and plasma exposure (scale 10 µm).

Weight loss due to the plasma bombardment was registered and erosion rate G was measured. Taking into account deuterium ion current to the surface erosion yield was evaluated. Fig. 4 shows erosion rate of the SEP NB-31 composite in plasma as a function of plasma current to material sample irradiated with high-energy ions and for comparison to the non-irradiated one. Erosion yield Y was deduced from these measurements as the slope of the curves presented on the picture. Though scattered data (for CFC), the result gives evidence that erosion rate is higher for irradiated (damaged) material. Enhancement factor of the erosion yield given by appropriate ratio of Y values for irradiated to non-irradiated materials. It was  $Y_{SEP irrad}/Y_{SEP} = 2.6\pm0.6$  for SEP NB-31 and even larger  $Y_{pyro irrad}/Y_{pyro} = 4.8\pm0.4$  for pyrograhite while the lower value  $Y_{MPG irrad}/Y_{MPG} = 1.6 \pm 0.4$  was found for MPG-8.



 $G, mg cm^{-2}h$ 

Fig. 4. Erosion rate of SEP NB-31 graphite vs plasma ion flux.

The results of double successive plasma exposure of carbon materials is illustrated in Fig. 5 showing erosion depth and erosion rate of three types of graphites under study after the first and after the second plasma exposures. All materials exhibit increase of erosion rate in the second plasma corresponding to the layer of maximal defect density involved in the erosion.



Fig 5. Erosion depth and erosion rate in double plasma experiment for 1 dpa samples of SEP NB-31, MPG-8 and pyrographite.

### 2.3. Tungsten

**Irradiation by**  ${}^{4}\text{He}^{+2}$  **ions.** Tungsten samples W (99.95% wt) 10×12×1 mm have been irradiated by  $\alpha$ -particles ( ${}^{4}\text{He}^{+2}$  ions) accelerated to 3.0-4.0 MeV. Three series of irradiations have been performed to reach high levels of radiation damage.  $\alpha$ -particles fluence values collected on the samples were 5.10<sup>17</sup> ion/cm<sup>2</sup>, 10<sup>18</sup> ion/cm<sup>2</sup> [7] and 3.10<sup>18</sup> ion/cm<sup>2</sup>. These values correspond to different levels of the obtained defects. Fig. 6 illustrates radiation defects production in tungsten by helium ions at energy of 4.0 MeV. The curve shown in the figure was obtained by calculation of the primary defect profile by the SRIM program [5] for 10<sup>17</sup> ion/cm<sup>2</sup> of helium ion fluence. The distribution of the radiation defects has maximum ~ 5 dpa at about 6.2 µm characterizing the range of the high-energy  $\alpha$ -particles in tungsten.

The study of the surface in the vicinity of irradiation area border on the tungsten samples has not shown as important deformation as on graphites. It was not detected with our profilometer on the first sample at  $5 \cdot 10^{17}$  ion/cm<sup>2</sup>, the two other samples have shown linear deformation effect of 0.1-0.3 µm or 2-5% as compared with 6 µm thickness of the damaged layer. On the third sample ( $3 \cdot 10^{18}$  ion/cm<sup>2</sup>) we observed a large area ~  $\frac{1}{4}$  cm<sup>2</sup> having experienced a very



FIG. 6. Calculated profile of radiation defects produced in tungsten ( $\rho = 19.35 \text{ g/cm}^3$ , 183.8 amu) irradiated by 4 MeV alpha-particles (He<sup>+2</sup>) to dose  $\Phi = 10^{17} \text{ a/cm}^2$ .



Fig. 7. Tungsten surface modification by high-energy  $\alpha$ -particles; a) the surface as prepared, b) the border of irradiation area on tungsten sample with the received ion fluence  $10^{18}$  ion/cm<sup>2</sup>: the left part was not irradiated; c) as irradiated with <sup>4</sup>He<sup>+2</sup> ions to fluence  $3 \cdot 10^{18}$  ion/cm<sup>2</sup>(scale bars in microns).

large deformation about 140  $\mu$ m but it was due not to swelling but splitting of the surface layer from the tungsten bulk took place. The surface of the sample after irradiation to 10<sup>18</sup> ion/cm<sup>2</sup> is given in Fig 7b. The picture was taken in the vicinity of irradiation border, which is well seen in the photo. Example of the sample irradiated with  $\alpha$ -particles to 3.10<sup>18</sup> ion/cm<sup>2</sup> is shown in Fig. 7c.

**Plasma effect on radiataion damaged tungsten.** Plasma exposure of irradiated tungsten samples has been fulfilled also step-by-step in order to determine erosion characteristics in the near surface layers. Deuterium ion flux on the surface was  $j=30-55 \text{ mA/cm}^2$  in these experiments, energy of ions was 250 eV (bias). Temperature of the samples during exposure was not higher than 40 C. Surface changes have been found after plasma bombardment of irradiated tungsten. An example of the radiation-damaged tungsten at  $10^{18}$  of  ${}^{4}\text{He}^{+2}\text{/cm}^2$  after plasma exposure to fluence  $3.7 \cdot 10^{21} \text{ D}^{+}\text{/cm}^2$  is shown in Fig. 8.



Fig. 8. Tungsten irradiated to  $10^{18}$  <sup>4</sup>He<sup>+2</sup>/cm<sup>2</sup> after plasma exposure to  $3.7 \cdot 10^{21}$  D<sup>+</sup>/cm<sup>2</sup> (at 250 eV) (scale 10 microns).

The successive plasma experiments were pursued till the depth of maximal defect concentration was reached. We have made 5 successive exposure tests of the sample having taken the  ${}^{4}\text{He}^{+2}$  fluence of  $5 \cdot 10^{17}$  ion/cm<sup>2</sup>. Total deuterium ion fluence on the surface from the plasma accumulated during this series was  $2 \cdot 10^{22} \text{ D}^{+}/\text{cm}^{2}$ ; and the surface layer eroded after 5 experiments was 7 µm thick (see Fig. 6). Considerable changes in the structure were observed when the sputtering process came to the layer of maximal defect concentration. Three parts of this sample are shown in Fig. 9: a) the no-damage zone subjected only to 5 plasma exposures, b) the border of irradiated zone (to the right), c) damage (irradiated) area. The defect structure is distinctly seen on the irradiated side.



Fig. 9. Tungsten surface after plasma exposure to  $2 \cdot 10^{22} D^+/cm^2$ : a) area subjected only to plasma, b) the border between the irradiated ( ${}^{4}He^{+2}$  fluence of  $5 \cdot 10^{17}$ , to the right) and non-irradiated zone, , c) damage (irradiated) zone (scale 100 microns).

Weight losses were measured in these tests and erosion rate was evaluated. It was found to fall in the range  $Y=(1-2.5)\cdot 10^{-3}$  at/ion as estimated for the sample having received  $5\cdot 10^{17}$   ${}^{4}\text{He}^{+2}/\text{cm}^{2}$ .

#### **3. Discussion**

The using of high-energy ions to produce radiation damage has revealed by these experiments to be very efficient as a method for investigation of fusion materials facing plasma. Radiation damage was reached at the level of displacement 1-10 dpa in average over the layer of ion penetration depth which was ~5  $\mu$ m in carbon materials and ~6  $\mu$ m in tungsten. The studied graphites and tungsten exhibited swelling effect after irradiation which was very large on carbons (4-fold on 10 dpa CFC ) and much lower on tungsten (2-5%) relating to the damage surface layer. Beside displacement and resulting damage during irradiation with fast ions those materials undergo sputtering. Surface modification due to these effects was detected on all materials. Splitting of the damage layer was observed on pyrographite and on tungsten. Cracks were found on 5 and 10 dpa MPG-8.

Taking into account the highly inhomogeneous distribution of the radiation defects produced in the surface layer the plasma exposure was made so that to have erosion layer-by-layer till the depth of maximal defect concentration is reached with intermediate analysis of the samples. By this experimental procedure the increase of erosion yield was detected on irradiated graphites when the maximum damage layer was open to plasma. Further development of the surface took place on the CFC, formation of holes, whiskers and cones was seen on the SEP and MPG. The comparison of erosion under plasma impact of irradiated carbon samples with those having no damage showed the higher values of erosion yield (by 2-3 for CFC, even by 4-5 for pyrographite). Our observations lead us to the conclusion that the radiation damage influences the plasma erosion of carbon PFM's appearing in its enhancement. The effect is supposed to be determined by the radiation damage accumulated in matrix that results in the material microstructure change from dense to more friable.

Damage of tungsten structure in irradiated samples has been revealed by SEM analysis under plasma flux. Nevertheless up to date the tungsten unlike the carbon materials has not shown distinct effect of damage on erosion rate in deuterium plasma and additional work and analysis are needed.

## 4.Conclusion

The presented experiments have been executed in the development of the new approach to the research of plasma facing materials for fusion aiming at the accounting for neutron radiation effect. Neutron damage was simulated on carbon materials and tungsten with high-energy ions accelerated on cyclotron. Irradiations of graphites CFC SEP NB-31, MPG-8 and pyrographite was made with 5 MeV  $^{12}C^+$  ions while 3-4 MeV helium ions  $^{4}He^{+2}$  were taken to produce damage in tungsten. The damage level of 1-10 dpa was reached on the materials. Swelling effect was registered on the irradiated samples and it was important on graphites.

The irradiated materials were subjected to deuterium plasma exposure in conditions simulating tokamak SOL in steady state at 100 eV of plasma ions on carbons and 250 eV on tungsten. The evidences of radiation damage influence on the erosion process have been found by analysis of deformation, surface modification and erosion data. Surface modification has been detected on the radiation-damaged materials. The study of erosion characteristics in plasma showed enhancement of erosion yield on carbon materials and structure damage on tungsten.

The new experimental approach developed in this work to explore the plasma-facing materials for accounting of neutron irradiation and the results obtained appear to be promising for the further studies of the combined plasma and neutron effect on fusion PFM's.

### 5.Acknowledgements

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