High particle and heat load material testing device

Ya Sadovskiy, L Begrambekov, P Shigin, A Ayrapetov, O Bidlevich, A Grunin, N Utkov
National Research Nuclear University MEPhI, Moscow, Russian Federation
E-mail: yaroslav@plasma.mephi.ru

Abstract. A brief overview of the high heat flux material testing facilities is presented which forms background for a new small-scale laboratory device capable of high heat flux material testing. The scheme of the device is presented and its working principles are described. High heat and particle loads are achieved by focusing of either electron or ion beam on the sample being tested. The sample is fixed to an actively cooled copper table. Experiments with hydrogen ion beam irradiation of an MPG-8 graphite sample are presented, in which heat loads of 270 MW/m$^2$ were achieved. SEM images of the sample surface after testing are presented.

1. Introduction

Ion-surface interactions are well studied, though the flux range used in most papers is $10^{19}$-$10^{22}$ ion/m$^2$s. It is well known that some processes strongly depend on irradiating flux intensity. For example, in paper [1] it is shown that chemical erosion of graphite decreases with increasing ion flux. This tendency is measured up to $10^{24}$ ion/m$^2$s flux, but not further. In paper [2] blisters were formed on tungsten under high flux hydrogen ion irradiation. The temperature of the sample was 480-520 K which is favorable for blister formation on copper, as blisters are formed at 0.3-0.4 melting temperature. Same comparison with copper can be made for flake formation and flake melting which also occurred at copper-related temperatures. This clearly indicates that during high flux irradiation properties of target material can change thus leading to effects previously unexpected.

These and other unforeseen effects would take place during the operation of the ITER first wall, and especially divertor. At the same time, there are quite few experimental devices capable of continuous irradiation of samples with fluxes higher than $10^{25}$ ion/m$^2$s. Some of these devices are either limited in variation of parameters or are narrow aimed in their purpose. Most of these devices are used for modeling of the conditions of ITER first wall and divertor, where heat fluxes of tens to hundreds of MW/m$^2$ are expected.

Quasi-stationary plasma accelerators, such as QSPA-T [3] or QSPA-Kh50 [4] are capable of very high pulse loads (in the order of GW/m$^2$), though the pulse duration is less than 1 ms and after each pulse the device needs “recharging” for several minutes. Thus, a usual material testing study involves up to 100 pulses. Contrary, typical ELM frequencies in ITER lie in the range of tens of Hz. High heat loads can be achieved with electron beams, such as utilized in JUDITH II [5] device. In this device a highly focused electron beam rapidly scans the sample area thus leading to a desired mean heat flux. In this case considering the heat load being stationary indeed is arguable. High plasma density sources are used in devices like Magnum-PSI [6], MARION [7] and PISCES [8]. These devices allow stationary testing high heat loads of up to 20 MW/m$^2$. Operation time is limited to several tens or hundreds of seconds. Moreover, in Magnum-PSI a combined stationary and pulse load operation...
regime is realized. All these devices are highly complex installations requiring a dedicated engineering infrastructure and staff to maintain and operate them.

At the same time, if one does not want to test a full scale ITER divertor tungsten mock-ups, a rather compact laboratory device, working with small samples is enough for researching surface processes occurring at high pulse and stationary heat loads. High heat load density can be achieved by focusing of an ion beam on the sample. Independent of the sample size, an active cooling system is required when working with high heat loads.

A device capable of stationary and pulse irradiation of samples by either ion or electron beam with heat flux density of up to 200 MW/m² is presented in the paper.

2. Experimental device

The scheme of the experimental device is presented in Figure 1. The device has a cylindrical vacuum chamber (1) with a cylindrical plasma chamber (2) inside. Basically the whole system has axial symmetry. A DC plasma discharge is initiated between a heated tungsten wire cathode (3) and ring-shaped anode (4). By changing of the cathode heating current the plasma discharge current can be varied easily. Typical discharge currents vary from 2 to 20 A. Cathode sputtering is minimized by using low discharge voltage in the range of 50-80 V. The anode is in fact two rings with a gap between them. Ions are drawn to the sample through this gap. The outer anode ring can be moved vertically by means of a motion feedthrough (5). Thus plasma edge geometry and ion beam shape can be varied. Residual vacuum in the device is better than 10⁻⁶ Pa. Basic components of residual vacuum are H₂O ≈ 75% and H₂ ≈ 25%. A differentially pumped quadrupole mass spectrometer is used for residual vacuum control, working gas purity control and leak finding. A lock chamber (7) is positioned at the bottom of the vacuum chamber allows quick sample exchange without the loss of the vacuum in the device. A gate valve (8) is closed when the lock chamber is at atmospheric pressure. A motion feedthrough with a PTFE sealing (9) is used to move the sample holder.

The sample (10) being tested is fixed to a sample holder (11) which moves on a polished stainless steel tube (12). The sample holder is basically a flat copper table which is actively cooled from the downside. To bias the sample an electric feedthrough (13) is installed in the vacuum chamber. When the sample holder moves into the vacuum chamber it contacts the feedthrough thus allowing its
biasing. The copper table is connected to the stainless steel tube through a cylindrical ceramic insulator, capable of >30 kV insulation. Dielectric silicon-organic fluid with low viscosity is used for cooling. This fluid circulates in a closed loop with a fluid to water heat exchanger in it. The copper table has radial fins on the downside to increase its surface area contacting the fluid. Copper table thickness, fin height and number of fins were optimized using numerical modeling. The whole system was designed to be able removing of the maximum available beam power. Sample temperature could be varied by putting special inserts between the sample and the copper table. Sample temperature is monitored by an optical pyrometer through the window on the top of the vacuum chamber.

All valves in the vacuum system of the device are pneumatic and thus can be activated remotely. Every operation on the device is automated and controlled through a LabView application specially designed for this device.

High voltage power supply was also specially designed for this project. It allows biasing the sample either positively for electron irradiation or negatively for ion irradiation. Maximum bias voltage is 25 kV. The power supply has a maximum power of 4 kW and can output this power at a wide range of voltages. For example: 200 mA at 20 kV, 400 mA at 10 kV or 800 mA at 5 kV. Continuous feedback for controlling of the voltage and current is realized by a dedicated PC with LabView RealTime software allowing up to 500 measurements per second. The voltage change over time can be pre-programmed thus allowing pulse irradiation with desired frequency and duty cycle. Pulse frequencies of up to 100 Hz are available. Pulse irradiation combined with stationary irradiation regimes are also possible.

The beam shape is governed by a number of factors: bias voltage, plasma density, relative position of the sample table and anode rings. By varying these factors the beam spot size on the sample can be changed. Thus, desired heat flux can be achieved independently of bias voltage. Maximum heat fluxed are realized when the beam is highly focused. One can estimate that in the case of a 5 mm diameter spot and 4 kW beam the average heat density would be 200 MW/m².

Secondary electrons are emitted from the sample as the ion beam strikes it. These electrons form a secondary electron beam going from the sample upwards. Accelerating these electrons draws power from the same high voltage power supply that biases the sample and thus decreases the ion beam power. Moreover, the secondary electron beam is so focused that it can damage elements of the vacuum chamber. Therefore, a ring shaped anti-dynatron electrode is installed above the sample holder. This electrode is biased so that it blocks the electrons emitted from the sample. The bias voltage of this electrode is higher in value than that of the sample. Shape and position of this electrode were numerically modeled to allow blockage of the secondary electrons with a least possible bias voltage.

3. Experimental results
The first primary experiments made on the device were performed with MPG-8 graphite samples. Hydrogen was used as a working gas. Various regimes of ion beam irradiation were conducted with continuously increasing heat flux. Heat flux was estimated by beam power and spot size on the sample. After exposing of the sample to a highly focused ion beam for 15 minutes a macroscopic crater was found on the sample. This crater was measured with a help of a stylus profilometer. Its diameter and maximum depth were found to be 3 mm and 50 μm. By knowing the shape of the crater conclusion on the beam shape could be made and flux density calculation could be refined. It was then calculated that at the centre of the beam the heat flux density reached 270 MW/m². The sample was not cooled during irradiation and its temperature was 1300 K. SEM images of the same spot on the sample surface after 5, 10 and 15 minutes of irradiation are presented in Figure 2 a,b,c.

Grain sizes of the MPG-8 graphite are in the range of 15-25 μm and they can be seen on the images. Also a more fine relief is seen on the surface of the grains. This relief has characteristic dimensions of about 1 μm. As the irradiation continues grains are sputtered and pores between the grains are opened. The fine relief on the grains is formed already after 5 minutes of irradiation and does not evolve over time. The sample was deliberately cracked in to pieces so that its cross section
could be examined. Well pronounced 15-25 \( \mu \text{m} \) grains were observed on the cross section with no signs of the fine relief. Thus a conclusion is made that this fine relief is a result of sputtering and redeposition processes at the surface and does not represent any structure in the bulk of material.

It is worth to mention that this kind of surface modification was observed at lower heat flux densities as well. Thus no effects special to high heat flux irradiation were found in this case.

![Figure 2. Same spot on the surface of the MPG-8 graphite sample after 5 (a), 10 (b) and 15 (c) minutes of highly focused hydrogen ion beam irradiation.](image)

4. Conclusions.
A laboratory experimental device capable of high heat flux irradiation of materials with either ion or electron beams is presented. The device is easy to operate and is fully automated, and allows pulse irradiation, continuous irradiation or combined pulse and continuous irradiation. By varying of the beam size heat fluxes of up to 270 MW/m\(^2\) could be achieved which is proved for a test experiment with an MPG-8 graphite sample. After the irradiation fine structure with a 1 \( \mu \text{m} \) grain size is observed on the surface of the 15-20 \( \mu \text{m} \) graphite grains.

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