Post Irradiation Testing of Materials for High Heat Flux Components of ITER

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1. Introduction

Thermonuclear fusion is considered as an inexhaustible energy source for future generations. ITER (International Thermonuclear Experimental Reactor), the next step fusion device which is based on the tokamak-principle will be designed to generate reactor relevant amounts of fusion energy (i.e. in the GW-range) from deuterium-tritium-reactions.

High heat flux components of ITER will be exposed to cyclic heat loads of up to 5 MW/m\(^2\) under normal and 20 MW/m\(^2\) under transient conditions. In order to remove these high heat loads, plasma compatible armor materials are required, which must be connected to a water-cooled heat sink. Candidate materials for high heat flux components are tungsten alloys and carbon fiber composites (CFCs), for areas of lower heat loads beryllium is foreseen. The heat sink shall be produced from the solution annealed copper alloy CuCrZr.

Under abnormal operation, the surface of the plasma-facing materials may be exposed to severe thermal shocks caused by plasma disruptions which may deposit a high energy of up to 100 MJ/m\(^2\) within a few milliseconds.

In addition to thermo-mechanical loads, the high heat flux components are exposed to 14 MeV neutrons generated in the fusion process. This neutron-irradiation may influence the properties of the materials and their joints.

Heat load simulation experiments on unirradiated samples have been carried out in several electron beam facilities before [1]. In order to study the degradation effects of neutrons, a first irradiation campaign for high heat flux components has been initiated in the High Flux Reactor (HFR) in Petten in 1994. It consisted of two parts:
2. Experimental Details

Actively cooled samples consist of a plasma-facing material (e.g. CFC, W or Be) and a heat sink material. Several joining techniques like brazing, hot isostatic pressing (HIP) or welding are used. Samples are tested by means of the electron beam facility JUDITH located in a hot cell at FZJ [3]. Special devices had to be developed for the installation and testing of the samples under remote-controlled conditions. Fig. 1 shows the principle and the technical data of JUDITH.

Due to the limited irradiation space in the nuclear reactor, long tubular connectors had to be avoided and a special clamping system was developed for cooling water supply of the mock-ups during the high heat flux tests (s. fig. 2) In this clamping system two tubes are pressed to the cooling channel of the sample. The vice is motor driven and the force is controlled by a load cell. Sealings between the tubes and the test sample are made from soft copper. For reasons of accessibility, the whole clamping system is mounted on the door of the JUDITH facility. After installation the door is closed and the sample is placed in an area covered by the electron beam.

Cooling during the tests was achieved by water of room temperature. The flow rate of cooling water was 12 m/s. Twisted tapes were used in order to improve the cooling effect.

The power absorbed by the mock-ups during the test was determined by water calorimetry. It is calculated from:

- PARIDE 1: temperature: 330 - 390 °C; cumulative dpa: 0.30 in beryllium or carbon, 0.18 in tungsten,
- PARIDE 2: temperature: 750 - 800 °C; cumulative dpa: to 0.35 in beryllium or carbon, 0.2 in tungsten,

Results of the post irradiation testing have been reported in [2]. In the meantime new production techniques for high heat flux components have been developed, and a second irradiation campaign was initiated in 1998. Again it consisted of two parts:

- PARIDE 3: temperature: ≈190°C; cumulative dpa: ≈0.2 in carbon, ≈0.15 in tungsten,
- PARIDE 4: temperature: ≈190°C; cumulative dpa: ≈1.0 in carbon, ≈0.6 in tungsten,
\[ P_{\text{abs}} = C_p \Delta T \phi \]

where \( C_p \) is the specific heat, \( \Delta T \) the temperature increase, and \( \phi \) the mass flow of cooling water. During the heat loading, the area covered by the electron beam is a little smaller than the total surface area. For the calculation of power density, the power was referred to the whole surface area. In [4] this problem was discussed in more detail.

In thermal fatigue tests, the mock-ups were heated for 10 seconds in each cycle (after this time at least 95% of the equilibrium temperature was reached); then they were allowed to cool down for another ten seconds. Ramp-up and ramp-down times were 0.5 seconds each. Normally 1000 heat cycles were applied at each power density.

Temperature distribution on the mock-up surface was controlled by an infra-red camera; a detachment of armor tiles is recognized as an increase of surface temperature. Additional pyrometers allowed the continuous supervision of surface temperatures during thermal fatigue tests.

3. Testing of Actively Cooled Samples

In general two designs are used for actively cooled components of ITER (s. fig. 3). The "flat tile design" consists of tiles made from CFC, tungsten or beryllium attached to a rectangular water cooled heat sink made from copper. In the monoblock design the CFC or tungsten tiles are drilled and directly connected to a cooling tube.

3.1 CFC Flat Tile Mock-Ups

CFC flat tile mock-ups have been produced from the silicon doped CFC material SEPcarb NS31 (\( \sim 8\% \) Si). In order to improve the contact area the tiles were laser structured. After coating with soft copper by the active metal casting technique (AMC) they were joint to a heat sink from CuCrZr by electron beam welding [5].

An unirradiated mock-up of this type was loaded for 1000 cycles at absorbed power densities of 11.5 and 20 MW/m\(^2\) without any indication of failure. But the failure limit was reached at 23 MW/m\(^2\) where the mock-up failed by progressive detachment of a CFC tile [6].
Two CFC flat tile mock-ups irradiated at 0.2 dpa and 1 dpa, respectively, were tested. A first screening at a low power density of only 1 MW/m² showed a huge increase of the maximum surface temperatures:

- Mock-up no.1: before irradiation: 154°C, after irradiation to 0.2 dpa: 250°C
- Mock-up no.2: before irradiation: 157°C, after irradiation to 1 dpa: 300°C

This effect is due to the dramatic reduction of thermal conductivity in carbon after irradiation [7].

Table 1 shows the loading history of the CFC flat tile mock-ups. At 15 MW/m² both samples did not show any indication of failure. During the whole loading process of 1000 cycles the maximum surface temperature is slightly reduced due to annealing processes in the high temperature areas of the mock-ups (s. fig. 4).

For the sample irradiated at 0.2 dpa, this annealing process is also observed at the first cycles at 19.5 MW/m² but after 200 cycles a beginning failure was observed in the infra-red image and the tile showed a progressive detachment.

A thermal fatigue test with the sample irradiated at 1 dpa was not possible. At 18 MW/m² the surface temperatures exceeded 2500°C and erosion of the surface due to sublimation was observed.

### 3.2 CFC Monoblock Mock-Ups

Former CFC flat tile mock-ups were produced by brazing with titanium. They showed a very good performance before and after irradiation [2].

New CFC flat tile mock-ups are produced in the following way [5]: CFC tiles from the 3D-material SEPcarb NB31 were drilled and after laser structuring they were coated with copper by the AMC technique. Then they were joint to the CuCrZr cooling tube by hot isostatic pressing (480°C, 100 MPa, 4 hours).

An unirradiated CFC monoblock produced in this way was loaded for 1000 cycles at 19 MW/m². At 23 MW/m² the experiment had to be stopped after 700 cycles because of heavy erosion on the surface. But no indication of failure of the CFC – Cu joint was found [6].

After irradiation, the surface temperatures for this type of mock-up increased even more than for the flat tiles. The maximum surface temperature for a CFC monoblock
irradiated at 0.2 dpa increased from 401°C to 1580°C at a relatively moderate power density of 5 MW/m².

Thermal fatigue tests with this mock-up were carried out at 10 MW/m² x 1000 cycles and 12 MW/m² x 1000 cycles without failure (s. tab. 2). But when the power density level was increased to 14 MW/m² heavy erosion occurred and no thermal cycling was possible.

3.3 Tungsten Macrobrush Mock-Ups

Tungsten macrobrush mock-ups were produced from chamfered W-1%La₂O₃ rods of 4 x 4 x 7 mm³. These rods were coated with OFHC-Cu and joint to the CuCrZr heat sink by electron beam welding.

Due to the high reflection of electrons in tungsten (45% compared to 5% in carbon), the maximum achievable power density on tungsten macrobrush mock-ups is 14 MW/m² (absorbed). An irradiated macrobrush mock-up did not show any indication of failure after 1000 cycles at 8 MW/m² and 1000 cycles at 14 MW/m² neither in the infra-red image nor in the post-mortem metallography. After irradiation however, the failure limits are reduced to 10 MW/m² approximately (s. tab. 3). Post mortem metallography shows a detachment of the tungsten rods at the border between W-1%La₂O₃ and soft copper (s. fig. 6) which is ascribed to an radiation induced embrittlement of the OFHC copper.

4. Thermal Shock Testing (Simulation of Plasma Disruptions)

Thermal shock tests were performed with different grades of carbon and W alloys. The kind of surface damage is dependent on the material.

− For metals (tungsten alloys, beryllium) the material is molten and with increasing power density the melt material is ejected or evaporated. After irradiation embrittlement a small contribution of brittle destruction may occur.
− Ceramic materials and carbons are destroyed by brittle destruction or sublimation (s. fig. 5).

Most samples in the experiments had dimensions of 12 x 12 x 5 mm³. They were loaded by thermal shocks of 5 ms length at energy densities up to 20 MJ/m². Heated surface areas were 5 x 5 mm² for carbon and beryllium, and 3 x 3 mm² for W respectively. In order to minimize surface conditioning effects, all samples were loaded
by repeated five shots. As all samples had to be installed by remote-control, a special sample holder was constructed [4].

Diagnostics in these experiments are:

− measurement of current through the sample to determine the exact deposited energy
− surface photography (by means of an image scanner),
− laser profilometry to characterize the depth and the shape of the generated craters,
− weight loss measurement to quantify the amount of eroded material

Fig. 7 shows as an example the comparison of unirradiated carbon materials with those irradiated in the PARIDE 1 and PARIDE 2 irradiation programs. All thermal shock tests have been carried out at 8.4 MJ/m². Materials under investigation were several 3D-CFCs (Dunlop Concept 1, Dunlop Concept 2, SEPcarb N112, SEPcarb NB31), a 2D-CFC (CX2002), a 1D-CFC (MKC) and the silicon doped 3D-CFC SepCarb NS11. In addition, the Ti doped graphite RGTi was tested.

In 1994, when the irradiation program PARIDE was started, carbon materials had a low priority for divertor applications and the number of available samples was rather small (2 to 3 per grade). Taking into account the large scatter in thermal shock tests with carbon materials, the statistics are poor. Nevertheless, some general conclusions may be drawn from these results.

In general, little differences in erosion are observed for the different materials and for the different irradiation conditions except for the silicon doped CFC SEPcarb NS11 which after neutron-irradiation shows a significantly higher erosion than all the other materials (this is understandable from the evaporation of residual silicon). Furthermore there seems to be a tendency for higher erosion for materials irradiated at 350°C in comparison to unirradiated samples and samples irradiated at 750°C. This is ascribed to the reduction of thermal conductivity in carbon materials irradiated at low temperatures of 350°C [7]. At the irradiation temperature of 750°C annealing becomes effective and the thermal conductivity comes near to the one of un-irradiated samples. Testing of a larger set of samples irradiated in the PARIDE 3 and PARIDE 4 programs is in progress.
5. Summary

High heat flux testing with CFC mock-ups is limited by the high surface temperatures due to the degradation of thermal conductivities of carbon after irradiation. CFC flat tile mock-ups and tungsten macrobrush mock-ups show a slight reduction of failure limits after irradiation which is attributed to irradiation induced embrittlement of the OFHC Cu. CFC monoblock mock-ups do not show any failure up to the maximum achievable power density.

The erosion of the different carbon materials during thermal shock testing (disruption simulation) is not very much affected by the carbon grade nor by neutron irradiation. But there is a tendency for higher erosion after irradiation at 350°C. This can be understood from the reduction of thermal conductivity at the lower irradiation temperature.
References


<table>
<thead>
<tr>
<th>irradiation condition</th>
<th>power density (MW/m²)</th>
<th>no. of cycles</th>
<th>result</th>
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<tbody>
<tr>
<td>unirradiated</td>
<td>11.5</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>23</td>
<td>450</td>
<td>progressive detachment</td>
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<tr>
<td>0.2 dpa (in carbon)</td>
<td>12</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>19.5</td>
<td>1000</td>
<td>progressive detachment (n &gt; 200 cycles)</td>
</tr>
<tr>
<td>1 dpa (in carbon)</td>
<td>11</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>18</td>
<td>(screening)</td>
<td>surface erosion</td>
</tr>
</tbody>
</table>

Tab. 1: Loading history of CFC flat tile mock-ups

<table>
<thead>
<tr>
<th>irradiation condition</th>
<th>power density (MW/m²)</th>
<th>no. of cycles</th>
<th>result</th>
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<tbody>
<tr>
<td>unirradiated</td>
<td>19</td>
<td>1000</td>
<td>test stopped due to heavy surface erosion</td>
</tr>
<tr>
<td></td>
<td>23</td>
<td>700</td>
<td></td>
</tr>
<tr>
<td>0.2 dpa (in carbon)</td>
<td>10</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>(screening)</td>
<td>surface erosion</td>
</tr>
</tbody>
</table>

Tab. 2: Loading history of CFC monoblock mock-ups

<table>
<thead>
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<th>irradiation condition</th>
<th>power density (MW/m²)</th>
<th>no. of cycles</th>
<th>result</th>
</tr>
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<tbody>
<tr>
<td>unirradiated</td>
<td>14</td>
<td>1000</td>
<td>no failure</td>
</tr>
<tr>
<td>0.1 dpa (in W)</td>
<td>10</td>
<td>1000</td>
<td>overheating, loss of tiles</td>
</tr>
<tr>
<td>0.5 dpa (in W)</td>
<td>10</td>
<td>1000</td>
<td>overheating</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>1000</td>
<td>loss of tiles</td>
</tr>
</tbody>
</table>

Tab. 3: Loading history of tungsten macrobrush mock-ups
**technical data:**
- total power: 60 kW
- acceleration voltage: ≤ 150 kV
- max. loaded area: 100 x 100 mm²
- scanning frequency: ≤ 100 kHz
- pulse duration: 1 ms ... cont.
- beam rise time: 130 ms

**diagnostics:**
- IR camera
- IR pyrometers: 200... 3000 °C
- video camera
- quadrupole mass spectrometer
- thermo-couples
- instrumented cooling loop

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**Fig. 1:** Electron beam test facility JUDITH

**Fig. 2:** Clamping system for water supply of actively cooled test modules
Fig. 3: Different designs of divertor modules for ITER

Fig. 4: Maximum temperature during thermal fatigue test of an irradiated CFC flat tile mock-up (irradiation condition: 0.2 dpa at 200°C)
Fig. 5: Mechanisms of erosion during plasma disruptions
above: metals,
below: brittle destruction of carbon materials
Fig. 6: Metallographic sectioning of an irradiated tungsten mock-up (1 dpa at 200°C) after testing under thermal fatigue conditions.

Fig. 7: Weight loss during thermal shock tests for different CFC materials.