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Neutron-irradiation effects on high heat flux components – examination of plasma-facing materials and their joints

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Abstract

The neutron-irradiation experiments PARIDE 1 and PARIDE 2 have been performed at 350°C and 700°C with fluences of 0.35 dpa. The major part of the post-irradiation tests are high heat flux simulation experiments carried out in the electron beam facility JUDITH. These tests cover thermal fatigue experiments with small-scale high heat flux components, and on the other hand, thermal shock tests on the plasma-facing materials. Actively cooled samples were made from CFC, or beryllium as plasma-facing materials and copper alloys as heat sink materials. Different designs (flat tile, monoblock) and joining techniques (brazing, welding) were used. Best performance was found for CFC/Cu monoblock mock-ups, but also the brazed Be/Cu flat tile mock-ups fulfill the operational requirements for first wall components. Thermal shock experiments show a higher erosion after neutron irradiation. This degradation is either due to a reduced thermal conductivity (carbon) or to a decreased ductility after irradiation (beryllium). © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

High heat flux components of ITER will be exposed to cyclic heat loads of up to 5 $\mathrm{MW/m^2}$ under normal and 20 $\mathrm{MW/m^2}$ 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, carbon fiber composites (CFCs) and (as a back-up) beryllium. 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 $\mathrm{MJ/m^2}$ within a few milliseconds.

Heat load simulation experiments on unirradiated samples are carried out in several electron beam facilities [1]. But 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. In order to study the degradation effects of neutrons, the neutron-irradiation experiments PARIDE 1 and 2 have been performed in the high flux reactor (HFR) in Petten, The Netherlands under the following conditions:

- PARIDE 1: neutron fluence: $0.28 \times 10^{25}\ \mathrm{m^{-2}}$ (according to 0.30 dpa in beryllium or carbon), temperature: 330–390°C.
- PARIDE 2: neutron fluence: $0.37 \times 10^{25}\ \mathrm{m^{-2}}$, (according to 0.35 dpa in beryllium or carbon), temperature: 750–800°C.

The irradiation rigs are contained in total 700 samples approximately. Experiments cover high heat flux testing, thermal shock testing, and testing of mechanical (tensile, creep) and thermal (thermal conductivity) properties. The largest part of post-irradiation testing is

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performed at Forschungszentrum Jülich (FZJ); thermal conductivity measurements are carried out at CEA, Saclay (France) [2].

2. Testing of actively cooled samples

Actively cooled samples consist of a plasma-facing material (e.g. CFC, Be, W) and a heat sink material. Several joining techniques were used, which are described below. All samples were tested by means of electron beam facility JUDITH at FZJ. The location of JUDITH in a hot cell offers the possibility to test neutron-irradiated mock-ups [3].

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. Pyrometers allowed the continuous supervision of surface temperatures during thermal fatigue tests. Flow rate and temperature increase during heat loading were monitored, and from these data an exact calculation of the absorbed power is possible.

2.1. CFC monoblock

CFC monoblock mock-ups have been produced from three grades of CFC armor material: SEPcarb N31, SepCarb N112 and Dunlop Concept1. Heat sink tubes were produced from the solution-annealed CuCrZr alloy and the dispersive-strengthened copper Glidcop Al25, respectively. Joining was performed through active metal casting (AMC) and brazing with titanium eutectic by Plansee AG [4].

In total six neutron-irradiated mock-ups of different combinations of CFCs and copper alloys and of both irradiation temperatures have been tested, first in screening tests and later in thermal fatigue tests. In order to avoid erosion of carbon, the surface temperature in these tests was limited to below 2500°C, and due to the reduced thermal conductivity a maximum power density between 12 and 16 $\mathrm{MW/m^2}$ (absorbed) was reached. After these screening tests, the power density was reduced by 10% and the samples were loaded by thermal fatigue for 100 cycles. No indication of failure was observed in these tests.

One of the samples neutron-irradiated at 350°C (Dunlop Concept 1/Glidcop Al25) later was loaded to 1000 cycles at 15 $\mathrm{MW/m^2}$ (absorbed). During the test, the surface temperatures were controlled by means of the IR camera. The resultant IR image near to the beginning of the test (cycle No. 8) and the temperature distribution along the plotted line are shown in Fig. 1. The corresponding temperature distribution during the 1000th cycle is shown for comparison. The higher temperatures near to the upper and the lower edges of the mock-up are due to the larger distance of these regions to the cooling tube. If the temperature distributions at the beginning and the end of the experiment are compared, it is observed that temperatures in the center stay stable, as those in the hotter regions on the upper and the lower edges decrease. This effect is ascribed to annealing in the CFC.

2.2. CFC flat tile

A CFC flat tile mock-up was produced through AMC technique by Plansee AG. The three 8 mm thick tiles from CFC SEPcarb N112 had been coated with OFHC copper and bonded to the CuCrZr body by electron beam welding [5]. Irradiation conditions for this mock-up were 0.35 dpa at 350°C.

Power density was increased in several steps each followed by 100 heating cycles up to 9.3 $\mathrm{MW/m^2}$ (absorbed). No indication of detachment was observed up to this heat load. But a slight over-heating was found on one corner of the center CFC tile. During thermal cycling at 13.5 $\mathrm{MW/m^2}$, the area of this over-heated region increased (Fig. 2) and after 50 cycles melting of copper was observed at the bond layer. After reduction of the heat load to values of 9.3 $\mathrm{MW/m^2}$, another 550 cycles have been applied to the mock-up. During this period the surface temperature distribution was stable and no indications of a further damage were observed.

During the post-experimental inspection, severe erosion was observed on the surface of the detached tile which indicates that the surface temperature locally must have been beyond 2500°C. This confirms the IR measurements of Fig. 2. Furthermore a small gap of 1 mm approximately was found between the CFC tile and the Cu heat [8]. But small threads of copper are observed bridging the gap. They were also found in postmortem metallography. Detachment and melting of copper has taken place during the 13.5 $\mathrm{MW/m^2}$ heating. After reduction of the heat load to 9.3 $\mathrm{MW/m^2}$ the copper threads were generated by re-solidification. During the final thermal cycling test they took over part of the heat transfer from CFC to the Cu heat sink, and hence no further detachment of the tile occurred.

2.3. Beryllium–copper mock-ups

Actively cooled beryllium flat tile mock-ups were produced from 8 and 3 mm thick beryllium tiles made from the grade S65C by brazing to a CuCrZr heat sink. The brazing process followed a proposal given by JET [6].

Three different types of actively cooled beryllium – copper mock-ups all irradiated up to 0.35 dpa at 350°C were loaded in thermal fatigue tests without any indication of failure:

- CuMnSnCe braze, tile thickness: 8 mm, 1000 cycles at ~6.5 $\mathrm{MW/m^2}$.

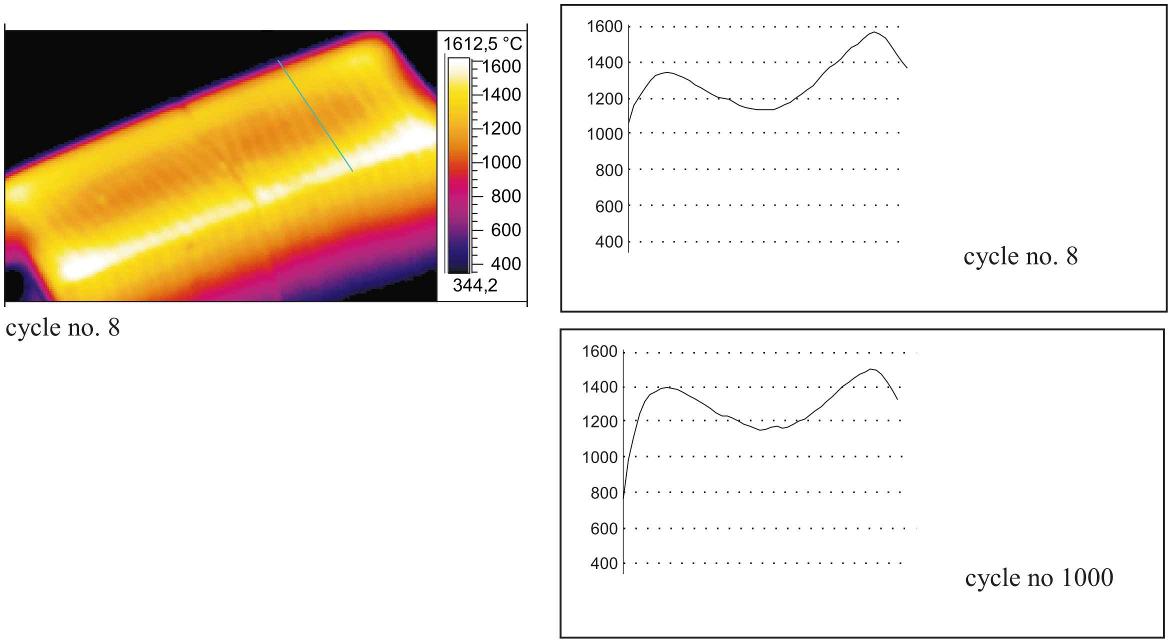

Fig. 1. Change of temperature distribution on the surface of a CFC mock-up Dunlop Concept 1/Glidcop Al25, irradiated at 350°C.

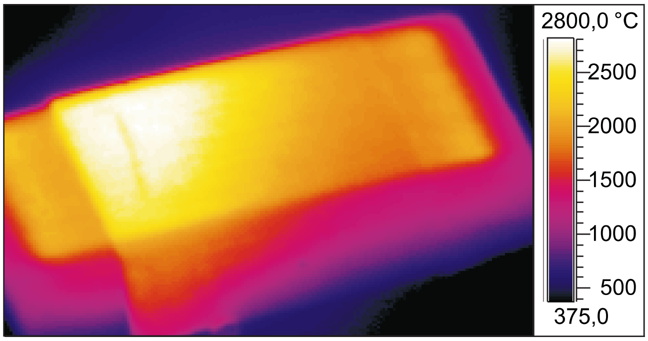

Fig. 2. Thermal fatigue test on a neutron-irradiated (350°C, 0.35 dpa) CFC flat tile mock-up made from SEPcarb N112 and Glidcop Al25.

- InCuSil braze, tile thickness: 8 mm, 1000 cycles at ~7 MW/m^2.
- InCuSil braze, tile thickness: 3 mm, 1000 cycles at ~9.5 MW/m^2.

In order to avoid evaporation of beryllium, in these tests surface temperatures were limited to 800°C. This means that heat loads were between 6.5 and 9.5 MW/m^2. More details on the course of these cycling experiments were reported in [7].

Metallographic inspections did not show any significant changes of the braze metals after neutron irradiation. For CuMnSnCe, a small intermetallic phase is observed in the middle of the braze layer for the unirradiated and the irradiated samples. No crack formation was found in the intermetallic. The braze layers appeared to be intact for both braze metals.

In addition to thermal fatigue tests, the two braze metals were characterized in shear tests. For CuMnSnCe, the shear strength decreased after neutron irradiation from 200 to 155 MPa, while for the InCuSil braze no irradiation influence was observed (300 MPa approx.).

3. Thermal shock testing

Thermal shock tests were performed with different grades of beryllium, carbon and W alloys. Most samples had dimensions of $12 \times 12 \times 5$ mm^3. They were loaded by thermal shocks of 5 ms length at energy densities up to 20 MJ/m^2. Heated surface areas were 5×5 mm^2 for Be and carbon, and 3×3 mm^2 for W, respectively. In order to minimize surface conditioning effects, all samples were loaded by repeated five shots. The diagnostics used in these experiments are described in [10].

In order to study the influence of neutron irradiation, all three sets of samples (unirradiated, $T_{\text{irr}} = 350°\text{C}$, $T_{\text{irr}} = 750°\text{C}$) were tested in the same testing campaign.

As all samples had to be installed by remote-control, a special sample holder was constructed [11].

3.1. Beryllium

The following grades of beryllium have been tested in thermal shock tests:

- PM beryllium grades: S65 (Brush-Wellman), TR-30 and TShG-56 (both delivered through Efremov Institute St. Petersburg, Russian Federation),
- condensed beryllium (Efremov Institute, Russian Federation),
- plasma-sprayed beryllium (Los Alamos National Laboratory).

The erosion for the first four materials characterized by weight loss measurements is shown in Fig. 3. The samples were loaded by five shots at about 15 MJ/m^2 each. All materials show a higher erosion by a factor of 1.5–2.5 after exposure to fast neutrons. The influence of the two irradiation temperatures however seems to be less significant. The results for plasma-sprayed beryllium are discussed separately in [11].

The degradation of thermal shock behavior is ascribed to an increasing brittle destruction after neutron irradiation [10]. This is in agreement with results from tensile tests which show an increase of brittleness after irradiation [12].

Parallel to the thermal shock tests, thermal conductivity measurements between room temperature and 700°C have been carried out with beryllium S65C by means of a laser flash apparatus. But no degradation was found after neutron irradiation. This indicates that the differences in thermal shock behavior must be ascribed not to changes in thermal but in mechanical properties.

Metallography of neutron-irradiated beryllium samples showed in general a similar behavior as the unirradiated samples before [13]. During thermal shock loading beryllium is molten and after re-solidification a columnar structure is found in the re-crystallized zone containing some cracks perpendicular to the sample surface. But after irradiation an enhanced crack sensitivity is observed for most beryllium grades [11]. Especially in the plasma sprayed grade an increase of both crack density and crack lengths is observed. Different to the other materials, in PS–Be the more severe cracks are parallel to the sample surface following the boarders of the spraying layers. Although the weight loss during thermal shock experiments for plasma sprayed beryllium is not dramatically higher than for the other grades, the cracks parallel to the surface may propagate during thermal fatigue experiments and may lead to a detachment of material.

3.2. Carbon materials

Thermal shock tests with unirradiated and irradiated carbon materials have been carried out at 8.4 MJ/m^2. 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 siliconized 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 no thermal shock samples have been foreseen in the program. The thermal shock tests shown in Fig. 4 have been carried out with thermal conductivity samples from PARIDE (thermal conductivity tests have

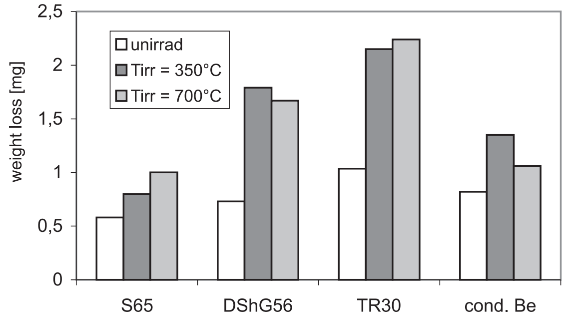

Fig. 3. Weight loss during thermal shock tests for different grades of beryllium.

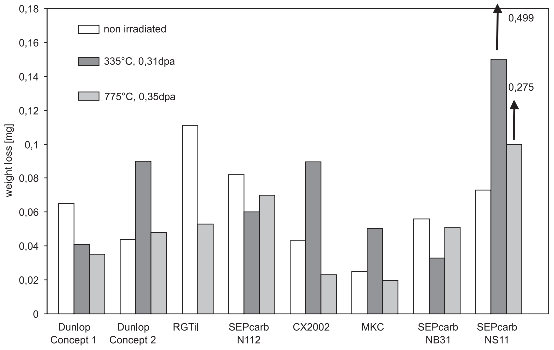

Fig. 4. Weight loss during thermal shock tests for different CFC materials.

been performed by Bonal [2]). But the number of available samples was rather small (2–3 per grade), and 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 siliconized CFC SEPcarb NS11 which after neutron irradiation shows a significantly higher erosion than all the other materials. 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 [2]. At the irradiation temperature of 750°C annealing becomes effective and the thermal conductivity comes near to the one of unirradiated samples. Metallographic investigations of thermal shock samples are in progress.

3.3. Tungsten materials

Several grades of tungsten have been irradiated in the PARIDE program: W-1% La_2O_3, W5Re, W30Cu and VPS–W. At the maximum energy densities available in the JUDITH facility (<20 MJ/m^2), the weight loss is in the order of the accuracy of the weighting system, except for W30Cu where Cu is evaporated. No significant influence of the neutron irradiation was observed so far.

4. Summary

CFC monoblock mock-ups produced by AMC and Ti brazing, show the best performance of all actively cooled mock-ups before and after neutron irradiation. A neutron-irradiated CFC flat tile mock-up produced by AMC and electron beam welding reached its limit at power densities of 10 MW/m^2 approximately. Brazed Be/Cu flat tile mock-ups fulfill the requirements of first wall application.

Both beryllium and CFC materials show some degradation in thermal shock behavior after neutron irradiation. For CFC at an irradiation temperature of 350°C the erosion is significantly higher than for unirradiated samples or samples irradiated at 750°C. For W alloys the erosion at energy densities up to 20 MJ/m^2 is low. No influence of the neutron irradiation was observed so far.

In the first quarter of 1999, a new neutron-irradiation campaign has been started. Irradiation conditions are 0.2 dpa at 200°C (PARIDE 3) and 1 dpa at 200°C (PARIDE 4). In this campaign new designs of high heat flux components [9] are tested.

References

[1] M. Rödig et al., in: Proceedings of the Fifth International Symposium on Fusion Nuclear Technology (ISFNT-5), Rome 19–24 September 1999.

[2] J.P. Bonal, CEA Report SEMI/LEMA/RT98-002/A, 1998.

[3] R. Duwe, W. Kühnlein, H. Münstermann, in: Proceedings of the 18th Symposium on Fusion Technology (SOFT), Karlsruhe (Germany), 22–26 October 1994, p. 355.

[4] G. Vieider et al., in: C. Varandas, F. Serra (Eds.), Proceedings of the 19th Symposium on Fusion Technology (SOFT), Fusion Technology 1996, Lisbon (Portugal), 16–20 September 1996, Elsevier, Amsterdam, 1997, p. 275.

[5] L. Moncel, J. Schlosser, R. Mitteau, L. Plöchl, in: Proceedings of the 20th Symposium on Fusion Technology (SOFT), Marseille, 7–11 September 1998.

[6] C.M. Ibbott, H.D. Falter, P. Meurer et al., in: Proceedings of the 18th Symposium on Fusion Technology (SOFT), Karlsruhe, 22–26 August 1994.

[7] M. Rödig, R. Duwe, J. Linke, G. Pott, in: 1998 Annual Meeting of the American Nuclear Society, 13th Embedded Topical Meeting on the Technology of Fusion Energy, Nashville, TN, 7–11 June 1998.

[8] B. Wiechers, M. Rödig, in: Conference on Thermal Stresses, Krakow (Poland), 14–18 June 1999.

[9] G. Vieider et al., in: Proceedings of the Fifth International Symposium on Fusion Nuclear Technology (ISFNT-5), Rome, 19–24 September 1999.

[10] J. Linke, H. Bolt, R. Duwe, W. Kühnlein, A. Lodato, M. Rödig, K. Schöpflin, B. Wiechers, these Proceedings, p. 1152.

[11] A. Lodato, M. Rödig, R. Duwe, H. Derz, J. Linke, R. Castro, A. Gervash, in: Proceedings of the Fourth IEA International Workshop on Beryllium Technology for Fusion, Karlsruhe, 15–17 September 1999.

[12] F. Moons, R. Chaouadi, J.L. Puzzolante, in: Proceedings of the Third IEA International Workshop on Beryllium Technology for Fusion, Mito City (Japan), 22–24 October 1997, JAERI-Conference 98-001, p. 241.

[13] M. Rödig, R. Duwe, A. Gervash, A. Khomutov, J. Linke, A. Schuster, in: Proceedings of the Second International Workshop on Beryllium Technology for Fusion, INEL-Report CONF-9509218, 1995.