

CERAMIC COATINGS FOR HTR GRAPHITIC STRUCTURES - TESTS AND EXPERIMENTS WITH SiC-COATED GRAPHITIC SPECIMENS

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Abstract

Graphite materials are used in High-Temperature Reactors for fuel elements and core structures. In the AVR and in the THTR it was successfully demonstrated that especially the spherical fuel elements showed excellent behaviour during normal operation and accident conditions. Improvements are possible as part of efforts to achieve catastrophe-free nuclear technology. In case of a massive ingress of air or steam into the primary circuit of an HTR, it is possible, if no active steps are taken, that serious corrosion of graphitic structures can happen.

For corrosion protection it is appropriate to provide these structures with ceramic (SiC) coatings. These coatings were produced by chemical vapour deposition and slip coating method. The coated graphitic specimens, spheres (without nuclear material) and other samples, were tested in many experiments, such as corrosion, mechanical and irradiation tests. The results of these tests show that SiC coatings applied to many graphites are corrosion-resistant and mechanically safe. The post-irradiation experiments showed for some coated graphitic spheres good corrosion properties at temperatures in the region of 750°C. For one material the corrosion resistance was even good for temperatures up to 1400°C(1600°C).

Furthermore, alternative forms of coated spheres, consisting of screwed half-shells, have already been tested successfully in corrosion and irradiation experiments.

1. Introduction

In HTR plants graphite materials are used for fuel elements and reflector structures. During the operation of HTR plants (AVR, THTR) these graphitic structures were extensively tested. Operating experience was very positive. It was proved that all operating demands (like high temperatures, neutron doses, mechanical loading) were fulfilled by the graphitic structures. It should be emphasized that the primary circuit of HTRs is very clean due to the good retention properties of the fuel elements, especially of the coated particles. Extensive tests proved that these good retention properties for fission products are valid even for temperatures up to 1600°C for several hundred hours. The development and testing of the spherical fuel elements can therefore be regarded as a complete success.

Although the above-mentioned properties of the graphitic structures are very good, research into improvements should continue. One of the HTR-specific accidents (analysed extensively in safety studies) is the entry of foreign media like air and water into the primary circuit. In design-basis accidents the corrosion of fuel elements is not critical for reactor safety. In certain cases of hypothetical accidents like massive air ingress it is possible that the matrix graphite of the fuel elements will be destroyed by corrosion in such a manner that a release of fission products may be possible. Up to now a large number of solutions have been considered to prevent such possibilities. For future nuclear plants new techniques have to be developed which will lead to a transparency of the technology applied. Therefore it is suggested that the outer surfaces of the graphite fuel elements should be provided with ceramic coatings.

The application of a ceramic corrosion protection coating is a very attractive solution not only for fuel elements but for graphitic structures like the bottom and side reflector too.

2. Graphite oxidation

Analyses in the field of graphite oxidation have been made in a large number of experimental tests and theoretical studies. To give an impression of how graphite spheres will be corroded by air, the results of experimental work in different facilities of the University of Duisburg [Epp 90], [Roe 94] are presented here.

In these test plants uncoated graphite spheres (without coated particles) of the original size (6cm diameter) were placed in silos and streamed by air from bottom to top. These tests were made in a temperature region from 600°C to 1200°C and for 3 different velocities. In Fig.1 the measured corrosion rates of these uncoated graphite spheres are presented. To give an impression of how large the corrosion protection by SiC coating on graphite spheres can be, the corrosion rate of a coated sphere is marked by the lower line in Fig.1.

3. Graphite materials

Different graphite qualities were coated with silicon carbide. To obtain a wide spectrum of information, nuclear and commercial graphites were used.

The nuclear graphite A3-3 is used as the matrix material for fuel elements in high-temperature reactors. This material is a composition of 64 wt% natural graphite, 16wt% petroleum coke graphite and 20 wt% resin binder. The fuel elements with this material are processed and heat-treated at temperatures below 2000°C. Therefore this matrix material is the only one which is not fully graphitized. [Jül 82]

The nuclear graphite material IG 110 is used for fuel element blocks in the Japanese HTTR plant. This IG 110 is a fine-grained isotropic graphite, which is fabricated from coke filler with binder on the basis of coal tar pitch. This graphite is isostatically pressed and fully graphitized (at 2800°C). [Jae 91]

The nuclear graphite ASR-1RS was used for reflector blocks in HTR plants. This material is a pitch coke graphite manufactured according to secondary coke technology; three impregnations with coal tar pitch; vibrationally moulded.

The nuclear graphite V 483 was employed for core-supporting columns in HTR plants. This graphite is a pitch coke graphite: fine grain, high binder content, isostatically moulded./Jül 87/

The commercial graphites are: FU 9512 (FP 379) from Schunk, EK98 and EK 432 from Ringsdorf and other graphites. These graphites are electrographites with filler material on the basis of pitch coke and binder on the basis of coal tar pitch./Sch₁ 89/, /Rin 91/

4. Production methods for ceramic coatings on graphite surfaces

The SiC coatings on graphite specimens (mainly spheres) were produced by two different methods: Chemical vapour deposition (CVD) and slip coating.

4.1. Coating methods based on CVD

Chemical vapour deposition methods are well known in chemistry and chemical engineering, so only a brief description will be provided here.

Chemical vapour deposition produces coatings from the gas phase. For SiC coatings, the SiC is deposited from the gas mixture $\text{CH}_3\text{SiCl}_3/\text{H}_2$ in a temperature range of 1150-1450°C at different pressure ranges. To obtain a continuous transition of the substrate and the coating material, an appropriate ratio of coating gas (CH_3SiCl_3) and reductant (H_2) is necessary as well as a suitable reaction temperature (approx. 1300°C).

Schunk /Sch₁ 89/ mainly supplied coated graphitic spheres of FU 9512 and IG-110 quality. In the present case a system with temperatures in the range of 1200°C is used. The SiC layers produced show a coating-thickness of 50-150 µm.

In addition, Schunk /Sch₂ 89/ provided coated spheres of A3-3 quality. The layers are produced by the following steps: 1) direct siliconizing of the graphitic spheres in a silicon steam atmosphere at temperatures below 1600°C. 2) immersion of the spheres in a SiC melt at temperatures up to 1500°C.

A great deal of interest has been shown also in CVD-SiC in functional gradient materials, whose composition changes continuously from SiC to C. In this case dense CVD-SiC with a thickness of 200 µm was formed on the graphite substrate and a CVD-SiC/C phase with a thickness of 1-2 mm containing voids was then formed on top of the first film. In this region, the ratio of SiC to C changes continuously and by adjusting the orientation of SiC it is possible to control the number of voids. Finally, a CVD-C film of a thickness of 100-200 µm is formed on top of the other films.

4.2. The slip-coating process

A new method for coating graphitic materials by SiC was developed at the Institute of Reactor Safety and Technology of the Technical University of Aachen (RWTH). This process consists in coating graphitic substrates by a slip and after that in a Si infiltration by a high-temperature process.

In a first processing step the uncoated substrate is wetted with a ceramic suspension consisting of α -SiC of different grain sizes, ultra-fine-grained graphite powder, organic binder and a solvent. After complete wetting of the substrate with this SiC slip the solvent evaporates out of the slip during a drying phase. A solid and a porous layer arise by getting out the solvent. The shares of binder polymerize and form a strong fixation between the solid phase of the slip and the surface of the substrate.

In a next step this specimen is put into a Si-contribution paste inside a furnace at a temperature of 1700°C. The melted Si (~1410°C) infiltrates the layer and the substrate of the specimen. During infiltration, the graphite of the outer surface and in the open porosity area at the inner surface, reacts with silicon to form β -SiC. At the same time, the silicon component reacts with the carbon components of the slip coat of β -SiC until the reaction is terminated. This new formed β -SiC grows on the α -SiC grains. The coating thus formed is a good connecting scaffold with the surface of the substrate. The layer penetrates into the graphite into a depth of nearly 1 mm. The open pores near the surface are filled with β -SiC. By this a strong adhesion is obtained.

The SiSiC coatings are produced with thicknesses of 75 to 150 μm . Successful coating was performed for nuclear graphites IG 110, V 483 and ASR 1 RS, as well as for electrographites IG 430, EK 98 and EK 432 by optimizing coating and siliconizing process performance.

5. Experimental results with SiC-coated graphites

To characterize the SiC-coatings produced, corrosion and thermal shock tests as well as ceramographic analyses are carried out. Some of these results have already been presented in former reports [Nea 97/, [Alk 98/. The new results of corrosion and post-irradiation experiments are additionally reported here.

The standard tests to check the corrosion resistance of the coated spheres were mainly performed at a temperature of $T=750^\circ\text{C}$ for an experimental period of 24 hours in a natural convection air stream. The aim of the development is to reach a corrosion rate of $R<0.1[\text{mg}/\text{cm}^2\text{h}]$ in the air stream for 200 hours in the temperature range from 400-1600°C. Corrosion tests in steam were performed in a temperature range from 600°C-1000°C. Additionally heated specimen were also shock-tested by dropping them into cold water. The analyses of these tests give information on the resistance to sudden changes of temperature of C/SiC structures. The following sections present experimental results for SiC coatings produced by different methods.

5.1. Corrosion results for coatings produced by CVD methods

5.1.1 Results for SiC-coated full graphite spheres

SiC coatings on graphite spheres by the CVD method have been mainly produced by Schunk [Sch,89/. We started with coating of spheres made of the graphite material FU 9512. This material has a thermal expansion factor which is similar to the SiC expansion factor. The results of the corrosion tests show very low corrosion rates ($R\leq 0.01\text{mg}/\text{cm}^2\text{h}$) for long test periods ($\leq 200\text{h}$).

Mechanical tests (dropped 10 times from a height of 2m on graphite spheres) were also performed on these FU 9512 coated spheres. No cracks or damage could be found. Corrosion experiments were then performed again on these spheres. Once again no damage could be found.

The aim of the development is to reach a corrosion rate of $R<0.1[\text{mg}/\text{cm}^2\text{h}]$ in air stream for 200 hours in the temperature range from 400-1600°C. This must be achieved for nuclear graphite materials.

SiC-coated spheres made from A3-3 graphite material showed an inadequate corrosion rate. Therefore we looked for other nuclear graphites to coat.

A very good corrosion-resistant quality was achieved with the IG 110 material, a graphite material used in Japan for HTR nuclear graphite. Corrosion experiments were performed with coated spheres of 60 mm diameter. The results were very good (Tab.1). Even short heat-up times of 3 hours had no influence on the results.

5.1.2. Results for SiC-coated screwed (hollow) graphite spheres

The main aim of our work is to obtain SiC coatings on A3-3 graphite spheres in the quality achieved on IG-110 graphite spheres. Until now the corrosion rate of SiC coating on A3-3-graphite spheres has not been sufficient. Therefore we tested alternative forms of coated fuel spheres. Five hollow spheres ($d_a=60\text{mm}$, $d_i>50\text{mm}$) made of IG-110 were produced consisting of two hemispheres screwed together (Fig.2). A3-3 spheres ($d<50\text{mm}$) without coated particles are installed inside two hollow spheres. The other three hollow spheres are without inner spheres. These screwed spheres were coated on the outer surface ($d_a=60\text{mm}$) with SiC and then tested for corrosion resistance see Tab.2. The corrosion-resistant screwed spheres were subsequently irradiated in the HFR Petten.

5.2. Corrosion results for coatings produced by the slip coating process

Tests for the corrosion resistance of the SiSiC-coated graphite qualities were carried out in an air atmosphere with an air flow velocity of 0.2 m/s at temperatures between 700°C and 1400°C for 24 hours.

The slip-coated samples of different graphite qualities showed no measurable corrosion due to graphite burn-up. The corrosion examinations were carried out at a temperature of 750°C-800°C for times ranging from 24 hours to 200 hours. At this temperature the graphite corrosion process is due to pore diffusion of oxygen through possible defects in the protective coatings. None of the samples examined at this temperature and times exhibited any measurable mass decrease, so that outstanding protection of the graphite samples can be concluded.

The reaction rates for slip-coated samples in air are very low for all electrographites and nuclear graphites (without A3-3). For instance the reaction rates for SiC-coated IG110 by slip coating have values in the region of $R\leq 0.01\text{mg/cm}^2\text{h}$. These results have already been presented in former reports /Alk 98/, /Nea 97/, /Mei 96/.

6. Irradiation and post-irradiation tests

To examine the integrity of different coatings after neutron irradiation, SiC-coated graphite spheres ($d=60\text{mm}+\delta_{\text{SiC}}$) were irradiated in HFR Petten. The temperatures and the neutron fluences for these irradiation tests were chosen in such way that the average operating conditions of an HTR-Modul were covered. The average surface temperature of the coated spheres ranged between 540 and 680°C, the neutron fluence ranged between 1.4 and $1.95\times 10^{25}\text{ m}^{-2}$ ($E>0.1\text{ MeV}$) Tab.3. Graphite spheres coated in different ways were supplied for this irradiation test. Three graphite materials were used for these spheres: IG 110, V 483 and A3-3. The SiC coatings were produced by the slip-coating process and CVD. Before irradiation, these coated spheres were tested for corrosion resistance for 50 hours at 750°C. In these corrosion experiments 5 coated spheres showed no mass loss. Only the coated A3-3 sphere had a small mass loss and was therefore not investigated further.

6.1. Irradiation rig D 247-01

Six coated spheres and one uncoated graphite sphere were loaded in an irradiation capsule in HFR Petten. This capsule consisted of a stainless containment housing the seven spheres in a graphite

structure see Fig3. The sample holder was instrumented with twelve thermocouples, three fluence detector sets and two gamma-scan wires /Con 96/

The irradiation test was performed in the HFR Petten for a period of four cycles or 93.89 full-power days.

6.2. Post-irradiation inspections

The weights of the spheres before and after irradiation are given in Table 4. Only sphere No. 6 is not taken into consideration, because the coating showed insufficient adhesion. The weight loss of the uncoated sphere (No. 1) is reasonable, because a borehole for temperature measurement was drilled into the sphere. The visual inspection in the HOT CELLS in Jülich shows that sphere No. 2 has two small points with peel-off. Spheres No. 3 and No. 7 have one place which looks different from the surrounding surface. The other coated spheres have no changes on the surface /Con 96/, /Der 97/.

To answer the question whether the coating of the irradiated spheres is damaged or not, we performed corrosion experiments in the HOT CELLS at Jülich .

6.3. Post-irradiation corrosion experiments

The corrosion experiments with these irradiated spheres were performed in the KORA apparatus in the HOT CELLS at Jülich.

6.3.1. Description of the KORA apparatus

The KORA furnace /Sch 99/, in which specimens up to spherical fuel element size can be heated, is installed in the gas-tight box of a HOT CELL. The resistance-heated furnace contains two concentric tubes placed inside each other, which may be made of fused silica, alumina or SiC, depending on the test temperature. The air first flows into the annular gap between the inner and outer tube where it is heated before reaching the specimen through an opening at the end of the inner tube (Fig. 4).

6.3.2. Results of KORA experiments

The KORA apparatus was used to perform temperatures from reactor operation (750°C) to accident conditions (1600°C). The heat up followed 200°C/h. During the test an air pressure of about 110 kPa and a flow of 30 ltr/h was reached. In contrast to higher temperatures, there is during the 750°C step no formation of a complete protecting oxide layer hindering SiC corrosion. Five SiC-coated spheres and also one uncoated graphite sphere were tested in the KORA apparatus. The results of these corrosion tests are summarized in Tab.5. Two of the five SiC-coated spheres (No.4 and 5) had no damage before and after the first post-irradiation corrosion tests at 750°C. Even the defective SiC spheres- No.2 and 7- had a significantly smaller corrosion rate at 750°C than the uncoated graphite sphere. One of the two spheres with no visible defect (No.5) had no weight loss after all three corrosion tests in air up to 1400°C. During the 1600°C-test the inner SiC furnace tube melted. Therefore the sphere was damaged by this occurrence. There are no indications that this sphere (if not damaged by the tube) would not pass the 1600°C corrosion test successfully.

6.4. Irradiation rig D 247-02

The performance of the irradiation for the second project was successfully completed in 1998 /CON 99/. The irradiation targets of the second project consisted of five screwed SiC-coated graphite spheres (see 5.1.2 and Tab.2) and three SiC samples each 15 mm in diameter and 16 mm in length. Three of the five screwed spheres were hollow without an inner sphere. The other two spheres had an inner sphere of $d < 50$ mm. The samples were irradiated in the HFR Petten under typical HTR-Module conditions between 600 and 770°C up to fast neutron fluence of $1.92 \times 10^{25} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). The main operating data were similar to the first experiment (rig D 247-01). A neutron radiograph image, taken shortly after completion of the irradiation, and the visual inspection after recovery of the samples showed no damage to the SiC coating.

The irradiated screwed spheres and SiC samples are now stored in the HOT Cells at Jülich. First measurements showed that the differences between the weights before and after irradiation are very small /Pot 99/ Tab.6 .

These irradiated coated hollow spheres will also be tested in the KORA facility in the next few months.

7. New SiC-coated IG-110 spheres

In order to improve the irradiation behaviour of the IG-110 coated spheres the coating techniques were slightly modified for CVD and for the slip-coating process. New coated full IG-110 spheres were produced and corrosion-tested (see Tab.7). These corrosion tested spheres will be prepared for the next irradiation rig.

8. Summary

In HTR plants graphite materials are used for fuel elements and reflector structures. As part of the efforts to achieve catastrophe-free nuclear technology it is appropriate to provide these structures with SiC coatings. These coatings were produced by chemical vapour deposition and slip coating method. The coated graphitic specimens, spheres (without nuclear material) and other samples, were tested in many experiments, such as corrosion, mechanical and irradiation tests. The results of these tests show that SiC coatings applied to many graphite materials (as electrographites and nuclear graphite IG110) are corrosion-resistant and can withstand the required mechanical loads. The post-irradiation experiments showed for some coated graphitic spheres good corrosion properties at temperatures in the region of 750°C. For one material the corrosion resistance was even good for temperatures up to 1400°C (1600°C).

The main aim of our work is to obtain SiC coatings on A3-3 graphite spheres in the quality achieved on IG-110 graphite spheres. Up to now the corrosion rate of SiC coating on A3-3 graphite spheres has not been sufficient.

Therefore we tested alternative forms of coated fuel spheres. One modification of the present fuel element concept is such that the fuel-free graphite zone of the fuel sphere consists of two screwed half-shells of IG 110 graphite instead of A3-3. Several experiments have been carried out for the coating and joining of such parts. A strong joint of the shells and corrosion resistance of the two parts can also be ensured for this case. Irradiation-damage of the SiC coatings was not observed.

The coating of full A3-3 spheres will be continued with different coating methods.

9. References

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Figures

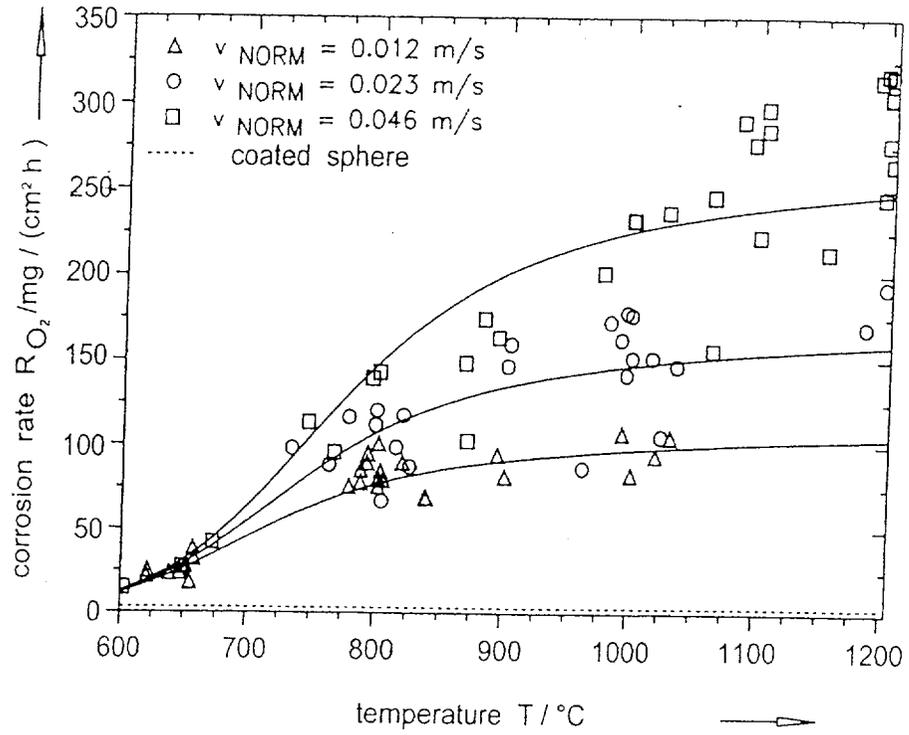


Fig.1: Corrosion rates of graphite spheres /Epp 90/, /Roe 94/

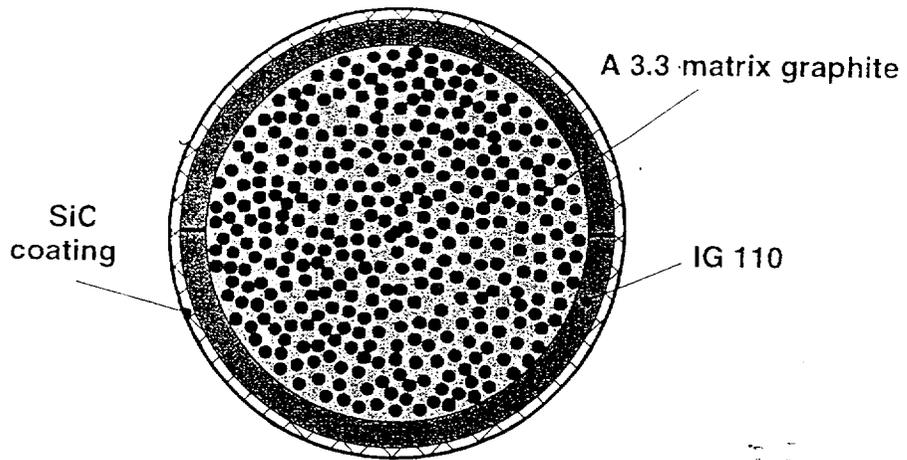


Fig.2: Proposal for a possible modification of the present HTR fuel element concept

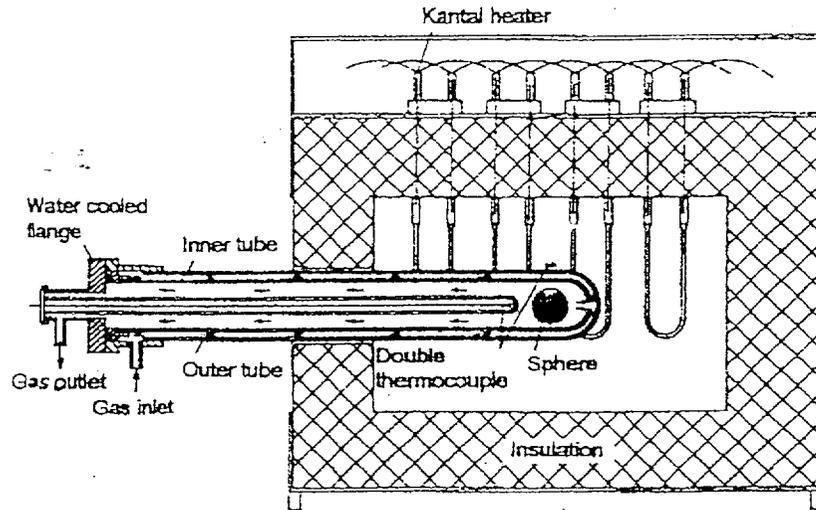


Fig.4: KORA resistance furnace

Tables

Tab.I: Corrosion tests of SiC-coated IG-110 graphite spheres(60mm) at 750°C

air streaming, 50 h total time			
graphite: IG 110		diameter: 6 cm surface:113cm ²	
sphere no.	heat-up time h	weight change mg	corrosion rate mg/cm ² h
1	3	+1	~0
2	3	-261	0.046
3	3	-13	0.0023
4	3	+1	~0
5	3	+10	+0.002
6	3	-0,7	~0

Tab.2: IG-110 screwed spheres, CVD-coated with SiC (2 with inner sphere, 3 without inner sphere)

Corrosion tests at 750°C, 50h, air stream results before irradiation			
hollow sphere	mass [g] before corrosion test	mass [g] after corrosion test	diameter [mm]
HK M1	200.089	200.088	60.3-60.4
HK M2	198.714	198.707 *	60.3
HK O1	86.730	86.729	60.3-60.4
HK O2	86.582	86.584	60.3
HK O3	87.338	87.341	60.3-60.4

*different balance after test

Tab.3: Cumulative neutron fluence data and full-power days, and averaged cycle temperature

Id no.	Item	Sphere no.						
		1	2	3	4	5	6	7
1	Irradiation duration Full-power days	93.89						
2	Neutron fluence E > 0.1 MeV 10 ²⁵ m ⁻²	1.0	1.39	1.69	1.9	1.95	1.83	1.47
3	Temperatures in °C, measured by thermocouples:							
	cycle 95.08	553	617	650	684	659	670	660
	cycle 95.09	523	580	638	674	670	691	672
	cycle 95.10	467	534	609	646	644	664	668
	cycle 95.11	538	596	645	672	665	678	652

Tab.4: Weight changes of the irradiated spheres

No.	Spheres	Weight		
		before irradiation [g]	after irradiation [g]	difference [g]
1	A3-3 reference	198.21	197.68	-0.53
2	IG 110 (4)	209.85	209.38	-0.47
3	IG 110 (4) CVD	204.04	203.91	-0.14
4	IG 110 (6) CVD	203.99	203.86	-0.13
5	V483 (2)	207.26	206.91	-0.35
6	No. A	not investigated		
7	IG 110 (1) CVD	205.04	204.84	-0.20

Tab.5: Results of KORA corrosion tests

Sphere	1	2	3	4	5	7
Graphite	A3-3	IG 110	IG 110	IG 110	V 483	IG 110
Manufacturing	no SiC		CVD	CVD		CVD
Visual inspection after irradiation		surface defect	small hole	intact	intact	small hole
Weight loss during test (g)						
750 °C, 20 h	53.1	2.6	34.3	0	0	9.5
1000 °C, 20 h	-	5.9	-	21.1	0	-
1400 °C, 20 h	-	14.0	-	-	0	-
1600 °C, 20 h	-	-	-	-	*	-

*damage of furnace tube and sphere

Tab.6: Weight changes of the irradiated screwed spheres

sphere	weight before irradiation [g]	weight after irradiation [g]
HK-M1	200.09	200.15
HK-M2	198.72	198.77
HK-01	86.78	86.78
HK-02	86.66	86.67
HK-03	87.34	87.43

Tab.7: Results of corrosion experiments at 750°C, 50h resp. 800°C, 12h in air streaming

ISR				
SiC-coated IG 110 graphite spheres (d=60mm) Si-infiltrated + CVD (Schunk) / 750°C, 50 h				
Sphere	Weight [g] of SiC-coated sphere	Weight [g] after 50h/750 °C in air	Δm [mg]	Diameter [mm] approx.
VK Si 1	212. 356	212. 353	-3	60.5-60.6
VK Si 2	211. 682	211. 683	+1	60.4-60.5
VK Si 4	212. 647	212. 655	+8	60.50
VK Si 6	211. 173	211. 186	+13	60.4-60.5
RWTH-Aachen				
SiC-coated IG 110 graphite spheres (d=60mm) slip-coated process / 800°C, 12 h				
VK-Trim14	206.30	206.30	0	60.35-60.45
VK-Trim18	206.85	206.85	0	60.40-60.42