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Graphite with SiC coatings

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Summary

This report contains state of the art information on SiC coatings for graphite materials. To obtain a wide information base, different graphite qualities, nuclear and commercial, have been used for the coating with SiC. Two manufacture methods for coating the graphitic specimens (spheres) with SiC are presented: Chemical Vapor Deposition (CVD) and paste siliconization coating method. The coated spheres (without fission material) were tested in mechanical-, corrosion- and irradiation experiments. The irradiation tests for coated spheres were performed in the reactor in Petten (Netherlands). The temperatures and neutron fluences for these irradiation tests were chosen in such a way that average operating conditions for an HTR Modul were covered. The results of the corrosion, irradiation and post irradiation tests for SiC coated graphite spheres show that SiC coatings applied to certain graphite materials are corrosion resistant and mechanically safe. Proposals for further development, especially for the matrix graphite, are mentioned.

It is the contribution of Forschungszentrum Jülich GmbH to deliverable D 2.5 WP3 of HTR-M1 (EC project: FIKI-CT-2001-00135)



Contents

1	Introduction.....	5
2	Corrosion of graphite.....	6
3	Graphite materials	7
4	Coating production methods.....	8
4.1	Coating methods basing on CVD	8
4.2	Paste siliconization method	8
5	Survey on Experimental Results with SiC-coated graphite specimens.....	9
5.1	Corrosion Tests	9
5.1.1	Corrosion tests in air.....	10
5.1.2	Corrosion tests in steam.....	11
5.2	Irradiation and post-irradiation tests	13
5.2.1	Rig D 247.01.....	13
5.2.1.1	Post-irradiation corrosion tests with D 247.01-spheres.....	16
5.2.2	Rig D 247.03.....	18
5.2.2.1	Post-irradiation corrosion tests with D 247.03-spheres.....	19
6	Proposal for further development.....	19
7	Conclusion	20
8	References.....	21
9	List of Figures.....	22
10	List of tables.....	22



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 components were extensively tested. Although the operation experiences with the large amounts of fuels used in HTR reactor operation were very well, the research for improvement should continue. Specific accidents for HTR are the ingress of air or steam into the primary circuit. In case of design-basis accidents the corrosion of the fuels and reflectors does not reach a safety critical condition. However, in the case of hypothetical accidents, if accident management actions are not possible, such events can lead to massive corrosion of reflector structures and graphitic matrix of the fuel elements. A release and eventually a damage of coated particles is then possible. The application of simple and easy safety principle consists in using such fuels which cannot be corroded by extraneous media. To avoid such damages by massive corrosion of graphite it is therefore suggested for future plants: Protection of the outer surfaces of graphite by SiC (silicon carbide) coatings. The main intention of SiC coatings is to realize a corrosion resistance for graphitic structures, especially for graphite fuel elements (spheres) in the range of operating and accident temperatures.



2 CORROSION OF GRAPHITE

In former R & D work it was shown extensively in which form and amount graphite will be corroded by air or steam. Especially the results of corrosion experiments with uncoated graphite spheres [1] will be presented here: In these experiments uncoated graphite (A3-3) spheres (without coated particles) of the original size (60mm 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 can be, the corrosion rate of a coated sphere is marked by the lower line in Fig.1.

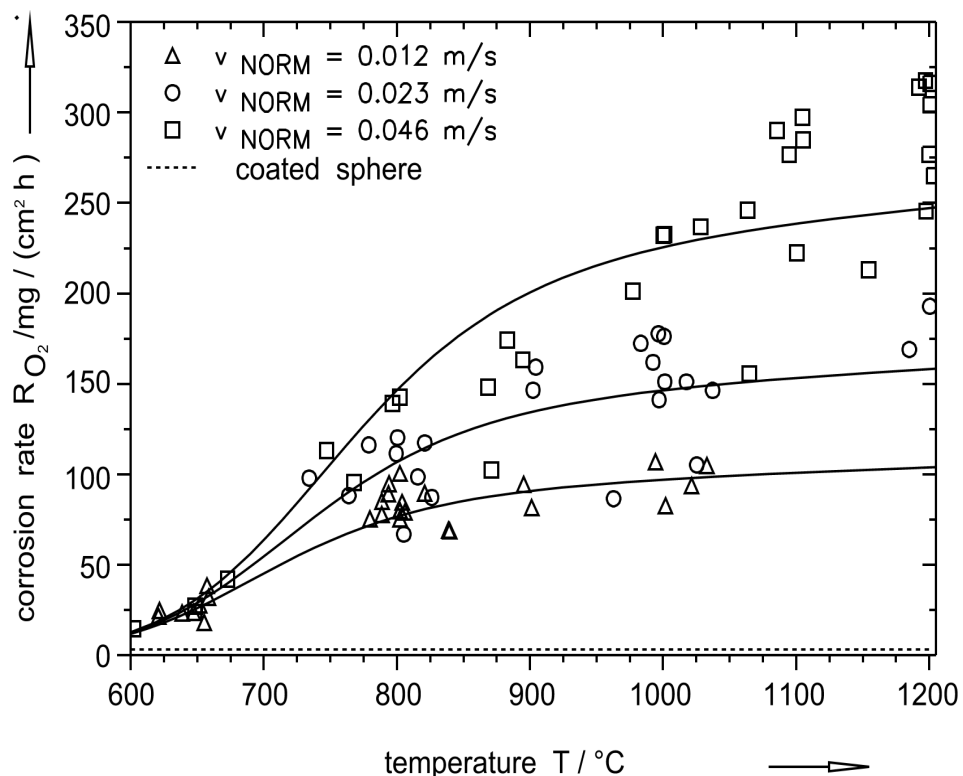


Fig.1: Corrosion rates of graphite spheres [1]



3 GRAPHITE MATERIALS

Beside the behavior of graphite in the presence of corrosion media, other qualities of different graphite materials can be important for the success of coating. To get a wide spectrum of information, different graphite qualities were used for testing of SiC coatings. Nuclear graphites as well as commercial graphite qualities were chosen. Tab.1 [2] gives a rough overview of the main properties of the used graphite materials.

properties		graphite qualities							
		nuclear graphite				commercial graphite			
		fuel element		structural material		FU 9512	IG 430	EK 432	EK 98
		IG 110	A3.3	ASR- 1RS	V 483				
bulk-density [g/cm ³]		1,77	1,73	1,78	1,75	1,8	1,82	1,73	1,85
young's modulus [kN/mm ²]	p	9,8	10,0	9,9	9,4		10,8	11,0	12,0
	v		9,7	9,2	9,3				
bending strength [N/mm ²]	p	24,5	34	26,0	30,8	40,0	37,2		
	v			23,0	30,4				
compressive strength [N/mm ²]	p	78,4	45	67,1	73,9	100	85,3	90	125
	v			63,1	74,9				
therm.expansion coefficient [10 ⁻⁶ /K]	p	4,6	2,89	4,7	4,1	4,7	4,8	2,9	3,4
	v		3,45	4,9	4,3				
therm. conductivity [W/mK]	p	116	70	125	109	75	139	100	80
	v		63	125	109				
concentration of ash [ppm]		< 10		390	520	300	< 10		< 100
porosity [Vol.-%]		21,6	20,45	17,37	20,55	10,0	20,12	20,22	17,50
producer		Toyo Tanso	Hobeg	SIGRI	Rings- dorf	Schunk	Toyo Tanso	Rings- dorf	Rings- dorf

p, v: parallel and perpendicular to the equatorial plane of the matrix sphere

Tab.1 Properties of the tested graphite qualities [2]

Most of the coating experiments have been made with the nuclear graphite material IG 110 which is used for the structures and fuel blocks of the Japanese HTTR core. The nuclear graphite A3-3 is used as matrix material for fuel elements in High Temperature Reactors. This material is a composition of 64 wt% nature graphite, 16 wt% petroleum coke graphite and 20 wt% resin binder. The processing and heat treatment of the fuel elements with this material is made at temperatures below 2000°C. Therefore, this fuel matrix material is not fully graphitized and requires specific developments for corrosion protection.



4 COATING PRODUCTION METHODS

Two different coating methods for producing SiC coatings on graphite specimens (mainly spheres) have been tested: Chemical Vapor Deposition (CVD) and paste siliconization method. Only a brief description will be provided here. More information can be seen in former reports [2], [3].

4.1 Coating methods basing on CVD

Chemical vapour deposition (CVD) is a well-known chemical proceeding for coating-technique, which produces coatings from the gas phase. There are two types of reactor-furnace for CVD: the hot-wall variety and, the cold-wall variety, which heats the base material directly in such a way that the reactor walls do not heat up. The hot-wall system is well-suited for coating a substrate with complicated shape, and is excellent for mass production.

CVD-SiC is used as a coating on a variety of shapes on order to improve resistance to heat and corrosion. It can be used on the surfaces of cylindrical and spherical base materials for use as heat-resistant tubes or corrosion-resistant components in energy plants.

Schunk Company [4],[5] produced CVD-SiC coatings mainly on graphitic spheres of FU 9512 and IG-110 quality (physical properties: see Tab. 1). Here, a hot-wall system with temperatures in the range of 1200°C to 1300°C and low pressure is used. The produced SiC layers show a film thickness of 50 - 150 µm. In addition, Schunk has coated spheres of A3-3 quality. The SiC layers therefore were produced in a different way:

- direct siliconizing of the graphitic spheres in a silicon steam atmosphere at temperatures below 1600°C
- immersion of the spheres in a SiC melt at temperatures up to 1500°C.

4.2 Paste siliconization method

This coating method was developed at the Institute of Reactor Safety and Technology of the Technical University of Aachen. The basic idea of this method is the homogeneous distribution of a defined silicon quantity on the graphite surface to be coated at adequate temperatures. During the thermal treatment the melted silicon reacts to β -SiC with the stoichiometrical amount



of carbon on the graphite surface. This results in the creation of a plastic formable silicon distribution paste, which is easy to apply to the substrate surface.

The silicon paste consists of fine-grained, pure silicon, an organic binder (wheat flour) and distilled water as the solvent. These components differ in their weight percentages depending on the graphite substrate quality and the thermal treatment parameters. Substrate porosity and thermal processing parameters, e.g. reaction temperature, heating duration, heating rates etc., are the main limiting parameters of the paste composition.

After mixing and homogenizing of the components the paste has a plastic consistency which allows application even on complex substrate geometries. This silicon distribution paste forms a direct connection to the substrate surface to permit siliconizing during thermal treatment. In a first step of the process, the organic binder is pyrolysed. Depending on the actual partial pressure, the liquefied or vaporized silicon is infiltrated to the substrate surface at a temperature above the corresponding melting temperature. A combination of a packed siliconizing process method provides the basis for a second method beside the direct silicon infiltration of the graphitic substrate (direct infiltration process): The SiC slip-wetted graphite specimen is treated by following siliconizing proceedings (slip coating process). The thermal processing of the direct infiltration is carried out at temperature between 1420 and 1750°C. Coating thickness varies between 50 and 150µm. The slip coating method produced coatings of 150 to 500 µm.

5 SURVEY ON EXPERIMENTAL RESULTS WITH SiC-COATED GRAPHITE SPECIMENS

The SiC-coated graphite specimens (mainly spheres) , produced by CVD- as well as by paste siliconization methods, were extensively tested by:

- Corrosion tests
- Irradiation tests
- Post-irradiation tests

5.1 Corrosion Tests

The standard tests to check the corrosion resistance of the coated spheres were 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 air stream for 200 hours in the temperature range from 400-1600°C.

Also corrosion tests in steam were made in a temperature range from 600°C-1000°C.



5.1.1 Corrosion tests in air

The following section presents some results of corrosion tests with coated specimens produced by different methods.

We started corrosion tests with SiC- coated graphite spheres (d=6 cm) made of the material FU9512 (Schunk). 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 for long test periods (Tab. 2).

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

air-ingress, 237 h total time			
graphite: FU 9512		diameter: 6 cm	surface:113cm ²
phase	heating time	weight-loss	corrosion rate
1	50	57,6	0,010
2	96	93,6	0,009
3	91	141,1	0,014
total	237	292,3	0,011

Tab.2 Corrosion tests of a SiC-coated FU9512 graphite sphere

The aim of our development is to get good corrosion resistance especially for nuclear graphite specimens. SiC-coated spheres made from A3-3 graphite quality showed an inadequate corrosion-resistance.

A very good corrosion-resistant quality was achieved with the well known nuclear graphite IG 110. First corrosion experiments were made with coated spheres of 45 mm diameter. The results were very good (Tab. 3). Also the results of corrosion experiments with coated spheres of 60 mm diameter, Tab.4, were very good. Therefore coated IG 110 spheres (d=60mm) were produced by different coating methods, tested on corrosion resistance and then prepared for irradiation in a rig in the reactor in Petten (The Netherlands).



air-ingress, 72 h total time			
graphite: IG 110		diameter: 4,5 cm	surface: 63,62 cm ²
phase nr.	heating time	weight-loss	corrosion rate
1	24	5,0	0,0030
2	48	4,6	0,0015
total	72	9,6	0,0020

Tab.3 Corrosion tests of a coated IG 110 graphite sphere (d=45 mm)

air-ingress, 50 h total time			
graphite: IG 110		diameter: 6 cm	surface:113cm ²
sphere nr.	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.4 Corrosion tests of coated IG 110 graphite spheres (d=60 mm)

5.1.2 Corrosion tests in steam

Besides corrosion tests in air, some coated specimens were examined in steam in the temperature range of 600-1000°C, vapor pressure 1-1.5 hPa, flowstream 0.7 kg/s.[2]



The examined slip-coated specimens showed no weight loss, but a slight increase in mass (Fig.2).

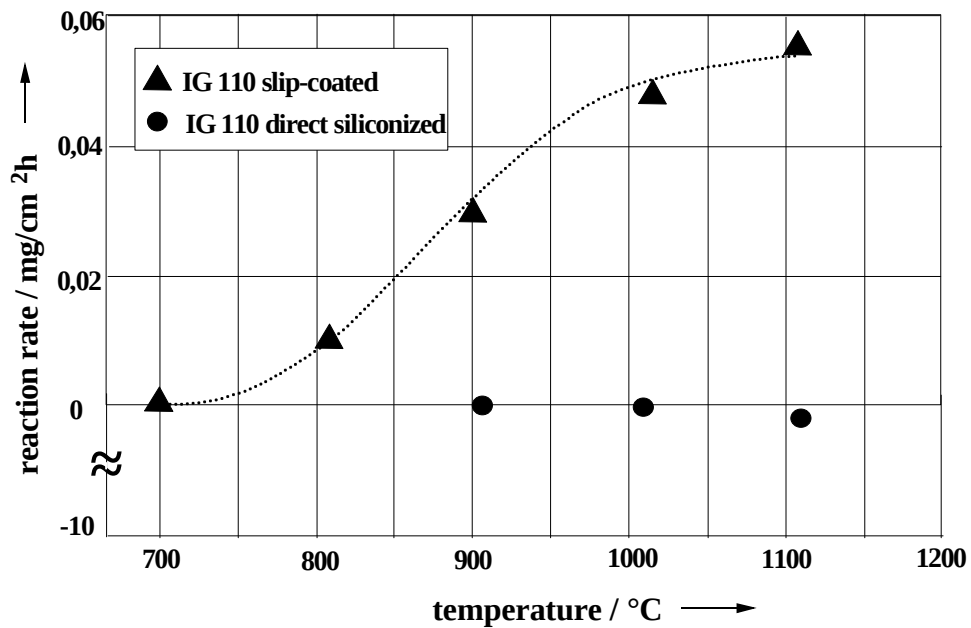


Fig.2 Corrosion tests of coated IG 110 graphite specimens in steam [2]

Additionally it is mentioned here that coated specimens with temperatures of 400 °C, 600°C and 800°C were tested in thermal shock tests into water of 18°C [2] . The coatings showed no damage after these experiments

Because the corrosion rate in air-stream is much higher (5x) than in steam the following tests were made for tests in air-stream only.



5.2 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 the High Flux Reactor (HFR) at Petten. The temperatures and the neutron fluences for these irradiation tests were chosen in such a way, that average operating conditions for an HTR-Module were covered. The coated spheres were installed in a irradiation capsule, Fig.3. This capsule consisted of a stainless containment vessel, housing the spheres in a graphite structure. The sphere holder was fitted with twelve thermo-couples, three fluence detector sets, and two gamma-scan wires [6].

5.2.1 Rig D 247.01

For this first irradiation-test coated graphite spheres, produced by different methods, were delivered. Three graphite materials were used for these spheres: IG110, V483 and A3-3. The SiC-coatings were produced by slip-casting, CVD and a special method for A3-3. Before irradiation these coated spheres were tested on corrosion-resistance at 750°C and 50 h. After these corrosion experiments three CVD-coated spheres, two slip coated spheres and one special coated A3-3 sphere were taken for the irradiation. For comparison one uncoated A3-3 sphere was added, Tab.5.

Specimen No	Identification	Diameter in mm			Weight in g
		Pol 'B'	Equator 'A'	Equator 'C'	
1	A3-3 reference	59.85	59.78	59.85	198.21
2	IG 110 (4)	60.48	60.53	60.50	209.85
3	IG110(4)CVD	60.19	60.13	60.17	204.04
4	IG110(6)CVD	60.32	60.38	60.21	203.99
5	V 483 (2)	60.43	60.20	60.25	207.26
6	Nr. A (TH)	61.11	61.18	61.41	218.56
7	IG110(1)CVD	60.24	60.32	60.32	205.04

Tab.5 Loading plan for rig D 247.01 [6]

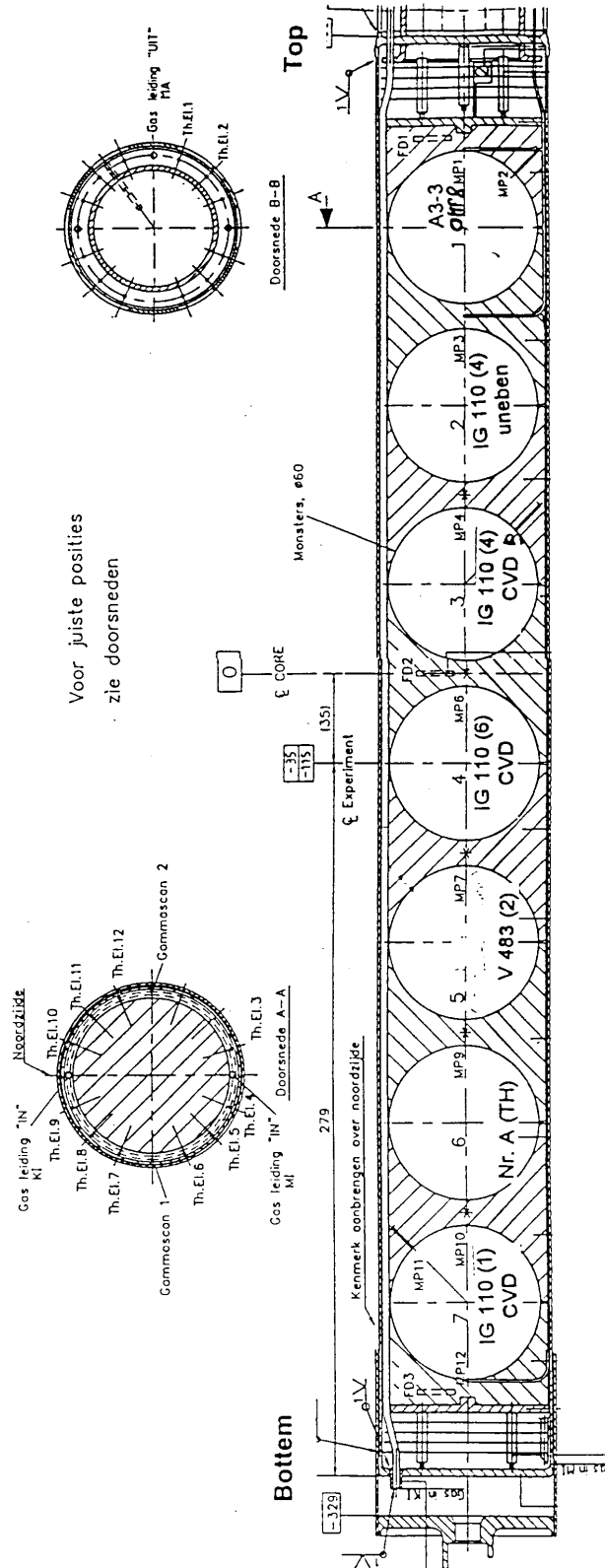


Fig.3 Schematic drawing of RIG D 247.01 [6]



The irradiation-test was performed during a period of four cycles (93.89 full-power-days). The average surface temperature of the coated spheres ranged between 540 and 680°C, the neutron fluence ranged between 1.4 and $1.95 \times 10^{25} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). Detailed data for the attained neutron fluence and the measured temperatures for each sphere are given in Tab.6.

Id no.	Item	Spheres no.						
		1	2	3	4	5	6	7
1	Irradiation duration Full power days	93.89						
2	Neutron fluence $E > 0.1 \text{ MeV}$ 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.6 Cumulative neutron fluence data and full power days, and average cycle temperature [6]

After the irradiation tests weight control and visual inspections of the coated spheres were made. The weight changes of the irradiated spheres are presented in the Tab.7. The irradiated coated sphere No 6 showed coating damages and was therefore not further taken into consideration. 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 shows that sphere No. 2 has two small points with peel-off. Spheres No. 3 and No. 7 have one place which looks different to the surrounding surface. The other coated spheres have no changes on the surface [6], [7].



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	Nr. A	is not investigated		
7	IG 110 (1) CVD	205,04	204,84	-0,20

Tab.7 Weight changes of the irradiated spheres of Rig D 247.01 [7]

5.2.1.1 Post-irradiation corrosion tests with D 247.01-spheres

To check the coatings of the irradiated spheres whether they are damaged or not, corrosion experiments were performed in the HOT CELLS at Jülich. A special furnace, called KORA [8], Fig.4, was used for these tests. This resistance-heated furnace consist of two concentric tubes. The air flows (30 ltr/h) 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. The five irradiated coated spheres and the one uncoated graphite sphere were tested in a temperature range from reactor operation (750°C) to accident conditions (1600°C). The results of these corrosion tests are summarized in Tab.8. 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.

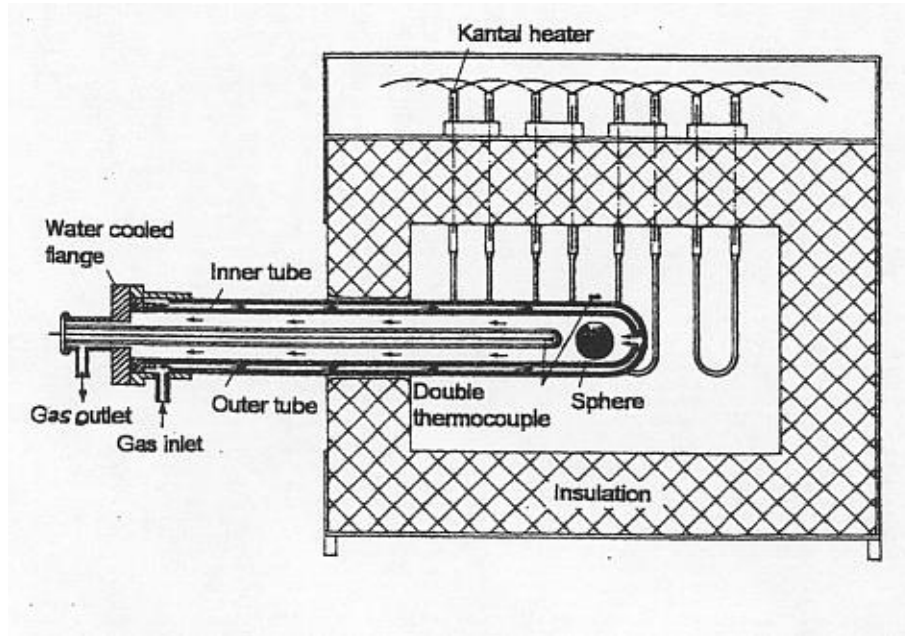


Fig.4 KORA resistance furnace

<u>Sphere</u>	1	2	3	4	5	7
Graphite	A3-3	IG110	IG110	IG110	V 483	IG110
Manufacturing	no SiC		CVD	CVD		CVD
<u>Visual inspection after irradiation</u>		surface defect	small hole	intact	intact	small hole
<u>Weight loss during test (g)</u>						
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.8 Results of KORA corrosion tests with D 241.01 spheres [8]



5.2.2 Rig D 247.03

In order to improve the irradiation behavior of the IG-110 coated spheres the coating techniques were slightly modified for CVD and for the paste siliconization process. New coated IG-110 spheres were produced and successfully corrosion-tested. These corrosion tested spheres were installed in a further irradiation rig D 247-03. The loading plan [9] shows 6 coated spheres, four (VK-Si) produced by CVD- and two (Trim) by paste siliconization method, Tab.9.

Specimen No	Identification Ref. [9]	Diameter [mm]	Weight [g]
1	VK-Si I	~ 60.5	212.36
2	VK-Si II	~60.5	211.72
3	VK-Si IV	~60.5	212.69
4	VK-Si VI	~60.5	211.22
5	Trim 14/90	~60.3	206.29
6	Trim 18/90	~60.3	206.86

Tab.9 Loading plan D 247.03

The D 247.03 experiment was performed in the HFR Petten similar to the first irradiation test during a period of four cycles with full power days of 99.1. The temperature profile ranged between 620 and 760°C and a axial neutron fluence profile between 1.12 and $1.64 \times 10^{25} \text{m}^{-2}$ ($E > 0.1 \text{ MeV}$). After visual inspection the coated spheres were transported to the HOT CELLS at Jülich. The weights of the irradiated spheres were determined, Tab.10 [10].

	VK-Si I	VK-Si II	VK-Si IV	VK-Si VI	Trim 14/90	Trim 18/90
Before irradiation [g]	212.36	212.72	212.69	211.22	206.29	206.86
After irradiation [g]	212.36	212.72	212.69	211.22	206.30	206.86

Tab.10 Weight changes of the irradiated spheres of rig D 247.03

Because the weight changes were small and the visual inspections of the irradiated spheres were not sufficient to determine the quality of the coatings, corrosion experiments were necessary.



5.2.2.1 Post-irradiation corrosion tests with D 247.03-spheres

The six irradiated coated spheres were tested in the KORA-facility at temperature of 800°C and 1400°C for 24 hours at a air flow of 30 [l/h]. Before these corrosion tests were started, sphere No. I and II were spot-checked by tests of fall, namely three times falling from a height of 50 cm (not higher possible in the glove box) on graphite spheres. No damages and weight loss were observed [11]. The results of the corrosion tests are presented in Tab.11.

Sphere No	Weight before test	Weight After 800°C	Weight After 1400°C	Weight Loss	Visual Inspections
VK-Si I	212.362	212.362	212.361	0.001	No damages, little changing of colour
VK-Si II	211.724	211.718	211.718	0.006	No damages, tiny bubble, little changing of colour
VK-Si IV	212.690	212.688	212.688	0.002	No damages, tiny bubble, little changing of colour
VK-Si VI	211.224	211.217	211.217	0.007	Small abrasion after 800°C, no damages, little colour changing
Tr 14/90	206.304	206.303	206.303	0.001	Tiny bubble and small pitting after 800°C, no damages after 1400°C
Tr 18/90	206.862	206.860	206.860	0.002	No damages, traces of white colour on the surface after 1400°C

all values in [g]

Tab.11 Results of KORA corrosion tests with D 247.03-spheres [11]

6 PROPOSAL FOR FURTHER DEVELOPMENT

The development of corrosion resistant SiC coatings on graphite spheres was successful for graphite qualities like IG110 and V 483. These coatings could be used for compacts fuels and reflector structures. Until now the corrosion rate for SiC coatings on A3-3 graphite spheres has not been sufficient. Therefore a modification of the present spherical fuel element was suggested in the way, that the fuel-free graphite zone is composed of two half-shells of IG110 graphite [2],



which could be coated very easily. This proposal should only be taken into consideration, if it is not possible in future efforts, to coat the A3-3 graphite quality. Therefore it is suggested to investigate into specific developments for A3-3 coating methods.

7 CONCLUSION

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 paste siliconization method. The coated graphite 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 coated graphite spheres of the quality IG110 and V483 good corrosion properties at temperatures in the region of 750°C 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 the efforts should be continued with different coating methods for the A3-3 graphite quality.



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9 LIST OF FIGURES

- Fig.1 Corrosion rates of graphite spheres
Fig.2 Corrosion tests of coated IG110 graphite specimens in steam
Fig.3 Schematic drawing of Rig D247.01
Fig.4 KORA resistance furnace

10 LIST OF TABLES

- Tab.1 Properties of the tested graphite qualities
Tab.2 Corrosion tests of a SiC coated FU9512 graphite sphere
Tab.3 Corrosion tests of a SiC coated IG110 graphite sphere (d=45mm)
Tab.4 Corrosion tests of coated IG110 graphite spheres (d=60mm)
Tab.5 Loading plan for rig D247.01
Tab.6 Cumulative neutron fluence data, full power days and averaged cycle temperature
Tab.7 Weight changes of the irradiated spheres of rig D247.01
Tab.8 Results of KORA corrosion tests with D247.01 spheres
Tab.9 Loading plan D247.03
Tab.10 Weight changes of the irradiated spheres of rig D247.03
Tab.11 Results of KORA corrosion tests with D247.03 spheres