

Aktuelle Konzepte für die Endlagerung hochradioaktiver Abfälle sehen vor, den Abfall in metallischen Behältern aus Stahl oder Kupfer zu verpacken und Untertage in Salz, Ton oder Granit als sogenanntem Wirtsgestein zu lagern. Der Zutritt von Wasser zu solchen Lagerstätten kann langfristig nicht ausgeschlossen werden. Damit kommt es zur Korrosion der Behälter.

Nach Ansicht von Experten wird die Funktionalität der metallischen Endlagerbehälter über 500 Jahre erhalten bleiben. Dies entspricht der geforderten Zeitspanne der Rückholbarkeit, aber keineswegs der Periode eines sicheren Einschlusses. Die Redundanz des Mehrbarrierenkonzepts – technische plus natürliche Barriere – geht damit verloren.

Vorgeschlagen wird, Siliziumkarbid als Einschlussmaterial in Form von Behältern, Behälterauskleidungen bzw. Ummantelungen zu verwenden. Siliziumkarbid, das auch in stellarer Materie nachgewiesen wurde, besitzt von allen bekannten Materialien die höchste Beständigkeit und ein ausgezeichnetes Rückhaltevermögen für die radioaktiven Spaltprodukte. Im Gegensatz zu Metallen, vor allen Dingen Kupfer, kann Siliziumkarbid in quasi beliebigen Mengen aus Silizium und Kohlenstoff enthaltenden Verbindungen unter Einsatz von elektrischer Energie synthetisiert werden. Es werden Konzepte für den Einschluss von abgebrannten Brennelementen aus Druckwasser-, Siedewasser- und CANDU-Reaktoren, von verglasten Abfällen aus der Wiederaufbereitung und von abgebrannten Brennelementen aus Hochtemperaturreaktoren vorgestellt.

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SiC encapsulation of high level waste for long-term immobilization

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1. Introduction

The safe deposition of high radioactive nuclear waste must be solved in the near future for 2 reasons mainly. Since the first operation of nuclear power plants several decades ago considerable quantities of waste have been gathered worldwide, but no disposal technology generally accepted is available so far in some countries [1, 2]. Furthermore, the acceptance of an optional use of nuclear energy in the future strictly depends on the solution of this problem as well. Despite differing opinions in the public on nuclear energy in general, the safe and well arranged disposal of waste is an undisputed matter and it may keep open presently unknown, beneficial options for future generations.

In the discussion about an appropriate repository for High Level Waste (HLW) two aspects are widely accepted today:

- land-based deep geological disposal and
- indefinite storage, but at least for 1 Mio. years.

In order to fulfil safety requirements for the biosphere, the multi-barrier concept – technical and natural barriers form an integrated whole – plays an important role. Whereas the natural barrier may be salt, clay or granite, the technical barrier consists of glass and metals, mainly steel and cast iron with spherical graphite. In Sweden, Finland and South Korea also copper and copper alloys are considered. Knowing that metallic canisters will lose their retention capability after a time which is short in comparison to the storage period, redundancy in the multi-barrier concept is lost quite soon. After 500 to 1,000 years the multi-barrier retention system is reduced to one barrier only – the host rock. Since there is no ideal host rock existing due to solubility or water ingress, the elementary safety principles in nuclear engineering, redundancy and diversity, are given up. For the long-term safety analysis an average lifetime of 500 years is presently proposed as

conservative value for the German Pollux container [3]. Therefore, much more attention must be paid to the long-term stability of the technical barrier.

The authors take the strict view that in a revalidation process of the present concepts and in open-result investigations of options for an optimal repository site

- the multi-barrier concept is mandatory and
- the long-term retention of the HLW can be provided only by an appropriate combination of the technical barrier with the host rock.

The importance of the canister must also be re-validated under the new German requirement of retrievability of the waste for 500 years at least.

The proceeding for the development of the new concept is based on the assumption that for safety reasons the existing waste remains more or less in the same temporary shape and packing as it is presently. This means in particular that

- fuel elements will be taken apart with separating the fuel rods from constructional parts, but without cutting the rods into smaller pieces and
- re-melting of vitrified waste into moulds of other dimensions is excluded.

Furthermore, all presently existing solutions for manipulators, canister transfer, drift and borehole disposal etc. should be kept and applied to any possible extent.

2. Fundamentals of ceramic encapsulation of high level waste

Considering that an ideal host rock does not exist, the long-term and safe retention of the radioactive fission products must be guaranteed predominantly by the technical barrier(s), whereas the natural barrier and the fact of deep geological disposal provide the required inaccessibility.

The technical barrier is not a uniform containment necessarily, but it may consist of different layers with their special

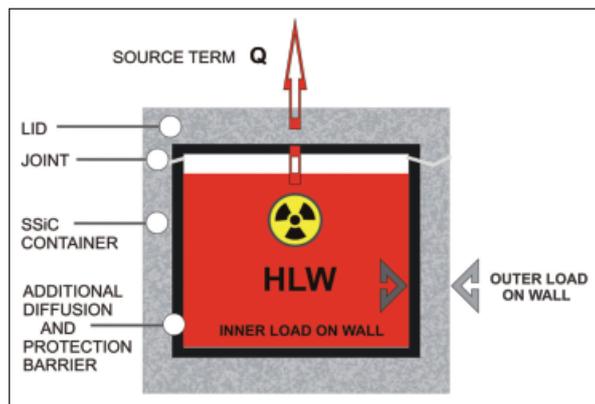


Fig. 1. Schematic of the inner technical retention barrier.

functions. The priority task of the inner container lies in the retention of the fission products; the outer encasements shall provide mainly mechanical stability necessary for handling, transport and absorption of static pressures and shear stress of the host rock. Solutions for the outer parts of the containments are existing [4, 5], for which reason this article concentrates in the following on the inner technical barrier with the retention function mainly.

In Figure 1 the inner technical barrier is shown schematically. The source term Q from this container should be as low as possible over a long period of time, 1 million years at least. The walls of the container must withstand different inner and outer loads which are listed in the following:

- Inner loads on canister wall: diffusion, radiation, heat, internal pressure, corrosive attack by fission products (silver and palladium e.g.).
- Outer loads on canister wall: corrosive attack by fluids, temperature, static pressure and shear stress by host rock.

From said loads the required properties of the canister material can be deduced such as: high corrosion resistance in acid and basic environment, high thermal conductivity, radiation resistance and gas tightness (no open pores).

A comparison of the properties of all questionable materials reveals that silicon carbide (SiC) is predestined for applications as inner canister material under extreme conditions. In the following chapter a more detailed description of SiC and its relevant properties is presented.

The retention capability of an un-cracked canister can be characterized essentially by three parameters: a) the breakthrough time t_b , b) the diffusion rate of the radioactive inventory through the canister wall and c) the corrosion rate of the canister wall.

The breakthrough time describes the initial phase of diffusion before equilibrium has been reached. In this sense t_b describes the maximal retention time of the barrier. The time t_b is given by

$$t_b = l^2 / 6 D$$

with the wall thickness l and the diffusion coefficient D of the respective nuclide in the barrier material. Breakthrough time t_b decreases with increasing temperature.

The source term Q is proportional to the diffusion rate:

$$Q \sim D / l$$

The diffusion rate and therefore the source term Q increase with rising temperature. In the ideal case the source term should be zero: $Q = 0$

In order to achieve a possibly low source term, or differently expressed, a long breakthrough-time, the container should be thick-walled and the diffusion coefficients of the fission products should be as low as possible. For a given and limited wall thickness it follows that the diffusion coefficients for select isotopes in the canister material are the determining factors for the breakthrough times.

To our present knowledge the diffusion coefficients in α -SiC of the long-living radioactive nuclei dominating the long-term radio-toxicity of waste such as uranium, plutonium and americium are unknown.

Data about the interaction between fission and activation products as well as oxide and carbide fuel with β -SiC has been reported from investigations of the TRISO coated particle behaviour at high temperatures ($>800^\circ\text{C}$) [6]. But for the conditions in a repository ($T < 200^\circ\text{C}$) and the proposed α -SiC canister the experimental data base is quite thin or non-existent. Nevertheless some general conclusions may be derived from [6] and other papers dealing with coated particle behaviour.

The diffusion coefficients of silver, caesium and strontium in silicon carbide are known from [7] and [8]. For a wall thickness $l = 10\text{ mm}$ SiC, breakthrough times of 100 million years for Ag and Cs respectively 10,000 years for Sr can be calculated and are graphically presented in Figure 2.

Pd diffusion probably occurs in both oxide and carbide fuel and also penetrates SiC grain boundaries. Ru and Rh have similar characteristics [9]. Out-of-reactor studies of fission product-SiC interactions in High-Temperature Gas-cooled Reactor (HTGR) fuel particles showed a penetration of Pd into SiC for UC_2 fuel. For temperatures above 800°C the Pd penetration and cross corrosion rate in SiC is given by $\log(\mu\text{m}/\text{year}) = 6.27 - 7140/T(\text{K})$ [10]. But the extrapolation of any of those high-temperature data to spent fuel conditions is conjectural [9]. Vekens and Foerthmann [11] investigated the transport behaviour of Pu and Am in low-enriched coated fuel particles at high irradiation temperatures (oxide, carbide and oxy-carbide kernels, 11 to 12 % FIMA, 1,100 to 1,600 $^\circ\text{C}$). In the SiC layer (35 μm thick) of the coated particles no transuranic elements were found. Ferguson and Walker [12] reported on the compatibility of β -SiC and uranium monocarbide in the range of 1,500 to 1,850 $^\circ\text{C}$. Above 1,600 $^\circ\text{C}$ a reaction occurred which appears to involve the formation of a molten shell at the original β -SiC-UC interface. It results in the diffusion of uranium into the β -SiC according to the relation $D = 0.01342 \cdot \exp(-68,000 \pm 20,00/RT)$.

According to Nabelek [13] the diffusion of U, Pu and other transuranic elements through SiC will not be the problem for the long-term deposition of spent fuel in SiC canisters, more important are the quality and corrosion stability.

In a most recent report Hunn et al. [14] describe the evaluation of as-irradiated

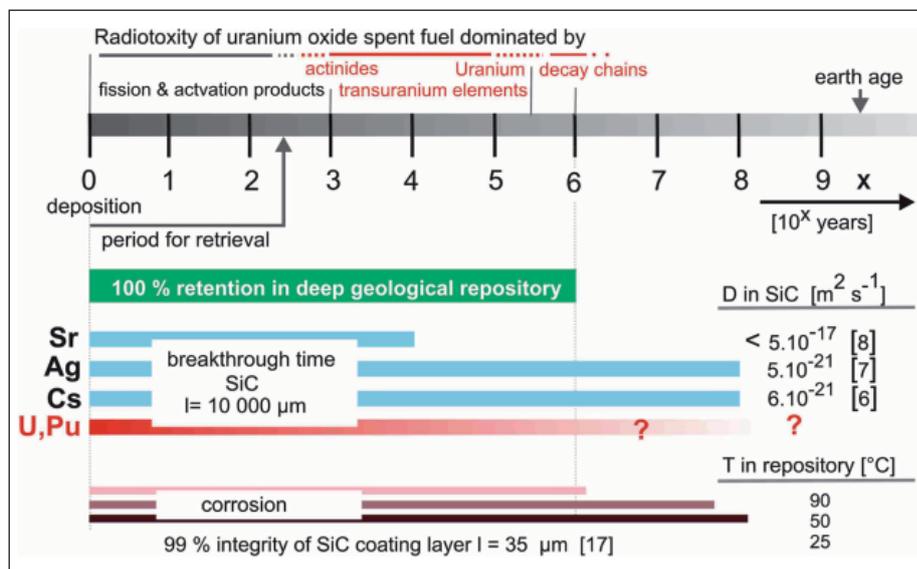


Fig. 2. Retentiveness of the SiC canister wall: diffusion and corrosion.

fuel performance (AGR-1 irradiated compact 6-1-1) using leach burn leach (LBL), IMGA, X-ray tomography and other material characterization techniques. The pre- and post-burn leach results were summed to obtain an inventory of fission products released by the SiC, but retained in the compact. The LBL analyses detected significant amounts of Ag, Eu, and Pd outside of the SiC. Less significant but measurable amounts of Sr, Ce, and Sm were also detected. Several other elements (e.g., Tc, La, Pr, and Nd) were detected, but the results were inconclusive because of the possible levels of background contamination in the hot cell. In general, there were no indications of physical irradiation-induced defects in the pyrocarbon or SiC layers.

Besides diffusion an alternate way for releasing radioactivity would be an extensive corrosion of the SiC canister wall. The characteristics and rates of reaction of rare earth bi-carbides with SiC in UC₂ fuel have been reported in [10, 15 and 16]. The corrosion occurs only by LaC₂ and NdC₂. Cross corrosion of SiC by La and Nd can be described by LaC₂: $\log(\mu\text{m}/\text{year}) = 3.44 - 4380/T(K)$; NdC₂: $\log(\mu\text{m}/\text{year}) = 2.52 - 2620/T(K)$. La and Nd do not corrode SiC in oxide or UO₂-UC₂ fuel because La and Nd exist as oxides. Once again, extrapolation to low temperatures, considerably lower than experimental, seems to be problematic [9].

The influence of corrosion on the integrity of TRISO particle coating during long-term storage in a salt mine repository has been investigated and discussed by *Nabielek* and *Verfondern* [17]. It turns out that, more than pressure vessel failure, it is the nature of the corrosion mechanism and the scatter in the corrosion law and SiC thickness that determines the onset of coating failure. The mean β -SiC coating thickness is 35 μm . Particle manufacturing defects and in-reactor failure fractions are at a level of 3×10^{-5} each. Assuming the parabolic corrosion law of SiC in granite water, the occurrence of a 1 % failure level during an accident scenario at a repository in salt is estimated at 1.7×10^6 years at 90 °C, 7×10^7 years at 50 °C and 2×10^8 years at 25 °C storage temperature (Figure 2). But a massively more extensive database is needed both for the model verification and the data validation for a reliable failure prediction in the repository in a deep geological formation.

To the present knowledge neither diffusion nor corrosion will deteriorate seriously the retention capability of robust SiC canisters.

As indicated in Figure 1, the retention capability can be improved by a special coating on the inner canister wall, which provides an additional diffusion barrier due to electrostatic repulsion as well as a corrosion protection barrier against Ag and Pd at the same time.

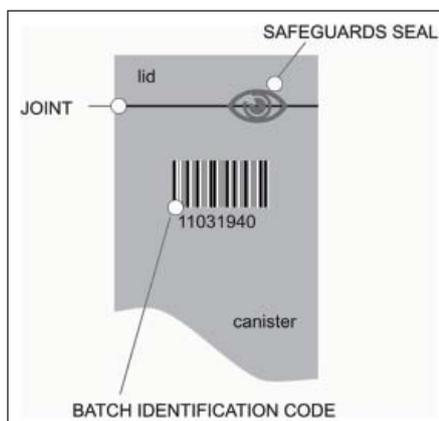


Fig. 3. Permanent canister identification by laser engraved codes and seals.

A durable canister labelling is required for safeguards reasons and the localization and content identification in case of retrieval. Laser engraving of seals and batch number codes ensure permanent canister identification (Figure 3).

3. Silicon carbide as encapsulation material

The inorganic chemical compound silicon carbide is extremely long-term stable due to its nearly ideal covalent bond. SiC occurs in stellar matter as well as on earth, known as the mineral Moissanite, but in vanishing small quantities only. The considerable technical demand for SiC in the areas of refractories, abrasives, ceramics

and special applications is covered by an industrial synthesis process using silicon and carbon containing compounds as raw materials and electrical energy for driving the endothermic reaction. The bulk of SiC on industrial scale is made from quartz sand and petrol coke. Besides the excellent material properties, summarized in Table 1, the abundant availability of raw materials make SiC the favourite candidate for nuclear waste encapsulation. If the metal copper shall be applied on a larger scale as planned today by a few countries only, the prices will raise astronomically for a material which is, today and in the future, necessary as electrical conductor.

Since the invention of the SiC sintering process in the early seventies of the last century [18] many progress has been achieved in fabricating different shapes and sizes of dense silicon carbide components. Despite many attempts of improvements, the preferable densification technology of today is still pressure less sintering at temperatures above 2,000 °C under inert gas atmosphere in batch or to a lesser extent in continuous furnaces. As standard sintering additives boron carbide (B₄C) and carbon (C) are applied, resulting in a one-phase material called “solid state sintered silicon carbide” (SSiC). The use of a combination of metal oxides, mainly alumina and a rare earth oxide, for promoting the sintering process [19] leads to a 2-phase material called “liquid phase sintered silicon carbide” (LPSiC) with some improvements in mechanical properties

parameter	symbol	unit	value(s)
density	ρ	$\text{g} \cdot \text{cm}^{-3}$	>3.1
open porosity	ϵ_o	[-]	0
Young's Modulus* [20]	E	GPa	420
Poisson ratio	ν	[-]	0.16
hardness** [20]	HV	GPa	26
fracture toughness	K_{IC}	$\text{MPa} \cdot \text{m}^{1/2}$	3.5
fracture strength (4-pt.-b.) at	σ	MPa	
20 °C			400
1,000 °C			450
1,500 °C			500
coeff. of lin. therm. exp. at	α	10^{-6}K^{-1}	
20 to 500 °C			2.75 to 5.05
500 to 1,000 °C			5.05 to 5.39
1,000 to 1,400 °C			5.39 to 5.65
thermal conductivity at	λ	$\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$	
20 to 500 °C			130 to 75
500 to 1,000 °C			75 to 45
1,000 to 1,400 °C			45 to 40
specific heat at	c_p	$\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$	
20 to 500 °C			700 to 1,050
500 to 1,000 °C			1,050 to 1,200
1,000 to 1,400 °C			1200

* $E(\epsilon) = E_0 \cdot e^{-2,15 \cdot \epsilon}$ with $E_0 = 420$ GPa

** $HV(\epsilon) = HV_0 \cdot e^{-6,0 \cdot \epsilon}$ with $HV_0 = 29$ GPa for SiC single crystal

Tab. 1. Selected properties of pressure less sintered, dense silicon carbide.

but clear deficiencies in oxidation and corrosion resistance.

One common characteristic of sintering processes lies in the linear shrinkage of the material in the range of 15 to 20 % which makes it more difficult to control the final dimensions and to avoid defects or even destruction with increasing component dimensions. The linear shrinkage and related problems do not occur in the case of silicon infiltrated silicon carbide (SiSiC), but the residual silicon in the finished component is responsible for strongly reduced temperature, corrosion and oxidation resistance.

Other types like re-crystallized silicon carbide (RSiC) or nitride bonded silicon carbide (NSiC) are not suitable for the envisaged application due to their open porosity and hence, poor corrosion and oxidation resistance as well as not existing retention capability.

Taking into consideration all the relevant properties, SSiC is the most appropriate material choice for encapsulation of high radioactive nuclear waste. Corrosion and oxidation resistance are at least by 2 orders of magnitude higher than for the other types of SiC.

Progresses in the shape forming technologies such as dry pressing, isostatic pressing, slip casting and injection moulding allow to produce various component shapes and sizes. The limitation in size as compared to metal components can be overcome by applying appropriate joining techniques, as proposed for two of the container designs described in the following chapter.

4. Type and construction of canisters

The types of the canisters to be applied for encapsulation of HLW are given by the shapes of waste generated by the different types of nuclear reactors and of re-processing plants.

As described in the introductory remarks already, the fuel elements of PWR and BWR have to be encapsulated in their full length of 4 meters approximately, because cutting the rods into shorter pieces would generate additional radioactive dust. In the present German reference concept it is intended to use steel cylinders with a lid [4], whereas the new concept plans to upgrade the steel by an in-liner of solid state sintered silicon carbide. A monolithic SSiC container of the required size is technically not feasible today for which reason inter-connected segments with a ceramic, so-called self-bonding technology are used. The schematic drawing of such a fuel rod canister is shown in Figure 4. Additional safety features which affect the mechanical strength of the arrangement, the retention capability, the neutron absorption and the corrosion resistance are described in the following chapter.

The nuclear waste of re-processing plants such as La Hague or Sellafield exists as vitrified body pored into stainless steel canisters of ca. 170 litres volume. Re-melting and filling into smaller canisters is not considered as an option due to the risk of releasing nuclear substances. For said reason it is proposed to encapsulate the existing canister by a segmented container of SSiC, as demonstrated in Figure 5. Due to its large dimensions, a monolithic SSiC vessel of the required geometry is not feasible today and therefore, the same bonding process as described for the fuel rod canister will be applied.

The spent fuel elements of CANDU-reactors are considerably smaller than the ones described previously and they can be packed into monolithic ceramic canisters directly. The canister is then hermetically sealed by the laser bonding technology described in [21] respectively by the self-bonding technology mentioned above. The arrangement of a CANDU fuel element in the SSiC canister is shown in Figure 6.

In Figure 7 the canister for spent fuel pebbles of high temperature reactors HTR (in Germany: AVR and THTR) is shown. Each container can enclose 3 graphite pebbles

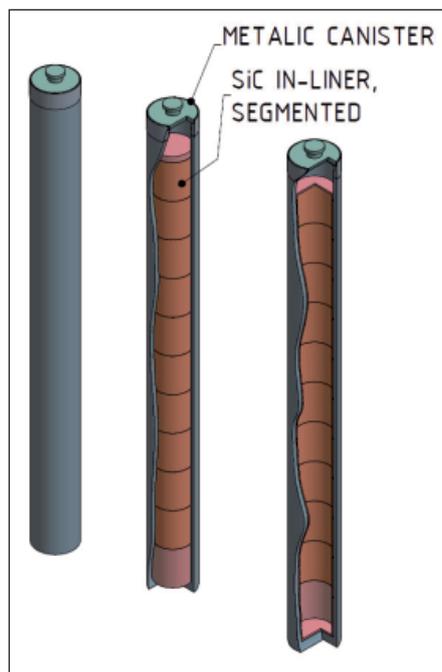


Fig. 4. SiC in-liner for spent fuel rods of PWR and BWR, overall dimensions: $D \approx 430 \text{ mm}$; $H \approx 4,980 \text{ mm}$; mass of SiC $\approx 190 \text{ kg}$.

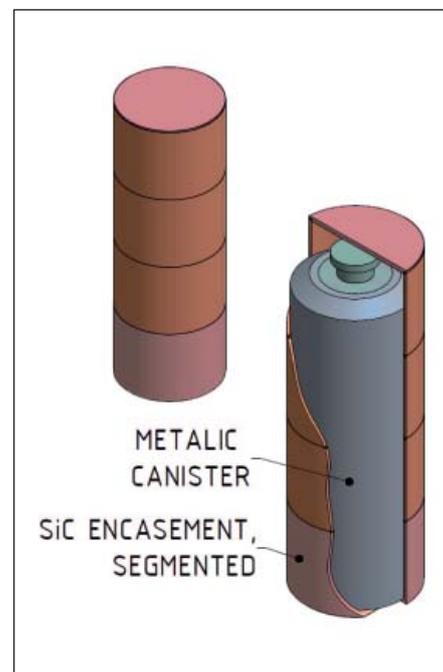


Fig. 5. SiC encasement for vitrified, high loaded waste, overall dimensions: $D \approx 465 \text{ mm}$; $H \approx 1,390 \text{ mm}$; mass of SiC $\approx 92 \text{ kg}$.

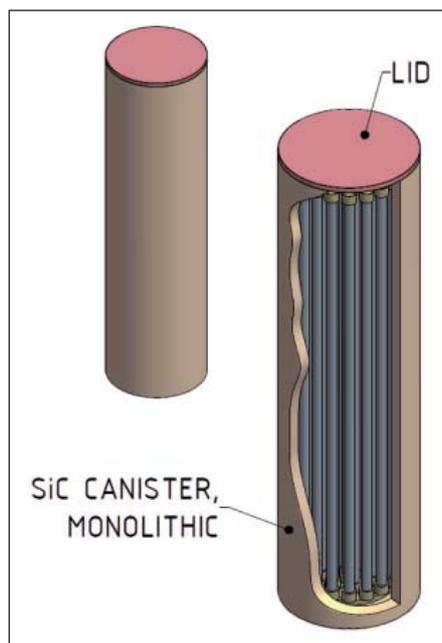


Fig. 6. SiC canister for spent CANDU fuel elements, overall dimensions: $D \approx 135 \text{ mm}$; $H \approx 530 \text{ mm}$; mass of SiC $\approx 8.5 \text{ kg}$.

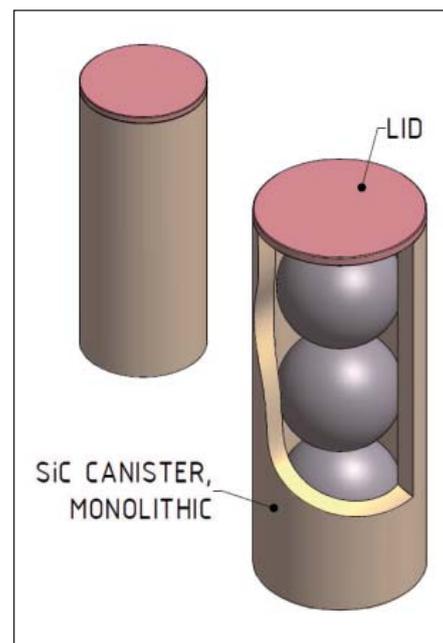


Fig. 7. SiC canister for spent fuel elements of HTR, overall dimensions: $D \approx 82 \text{ mm}$; $H \approx 205 \text{ mm}$; mass of SiC $\approx 1.7 \text{ kg}$.



Fig. 8. SiC pilot canister with 3 graphite pebbles, overall dimensions: $D \approx 82 \text{ mm}$; $H \approx 205 \text{ mm}$.

with the so-called TRISO particles inside as well as capsules or compacts applied elsewhere as fuel elements for HTR.

The photos of the Figure 8 show demonstration canisters of SSiC with a wall thickness of 10 mm. The production technology for this type of encapsulation containers on an industrial scale is readily developed.

5. Upgrading canister properties by potting

With the exception of the canisters for vitrified waste (compare Figure 5) all the other canisters exhibit some void volumes after filling, either between the rods or the pebbles. Before closing the canister those voids can be filled-up with other radioactive material (C-14 contaminated reactor graphite e.g.) as heat spreader. But there are several good reasons for the use of a specially tailored potting compound: to increase the mechanical stability of the whole arrangement and to improve the retention capability. Such a situation is shown schematically in Figure 9 for the fuel rod canister.

The potting compound itself can either be a dry flowable mass consisting of silicon carbide and/or graphite particles or a liquid with high solids content. The last-mentioned option offers the advantage that the potting compound can be composed in such a way to generate a 2-phase ceramic material after drying and reaction, induced by radiation and heat from the spent fuel. The drying process must be fully completed before closing the canister whereas the radiation induced reaction may occur over a longer period of time, resulting in the composite material silicon carbide/carbon. The left part of Figure 10 shows a schematic representation of the circumstances between

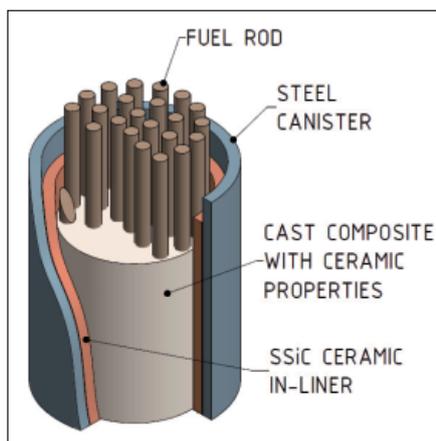


Fig. 9. Detail of a canister with spent fuel rods in potting material.

the fuel rod and the canister wall with the formation of a glassy carbon like layer on the inner wall side. Despite its high porosity the composite exhibits considerably high mechanical strength and provides additional retention spaces in the filler matrix be-

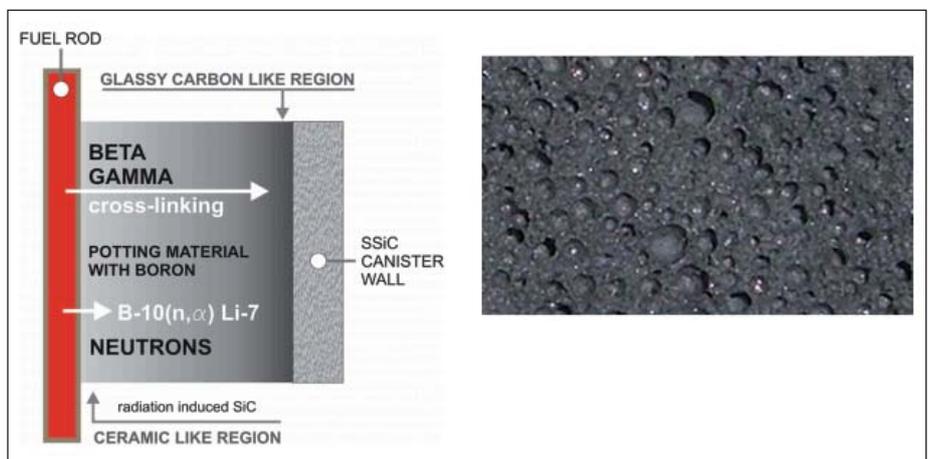


Fig. 10. Cross-linking of potting material and generation of radiation induced SiC under radiation and heat of spent fuel elements.

tween the rods. The photo in the right part is suggestive of the appearance of the fully reacted potting compound with typical pores of approximately 0.2 to 1 mm in diameter. Furthermore the composite will reduce the source term Q drastically even in the loss of integrity of the in-liner or monolithic SSiC canister caused by shear stress (faults or geological shifting in the host rock).

For several reasons boron carbide is added to the potting material. On one side boron maintains subcriticality in the repository, on the other side it plays an important role in developing the composite structure (providing the necessary activation energy for the Si-C exothermic reaction via the high LET radiation from the $B-10(n, \alpha) Li-7$ reaction). By absorbing neutrons, boron is also an effective radiation shield for the SSiC canister minimizing radiation damages in the wall.

6. Conclusions

The encapsulation of nuclear waste – especially of HLW – in SSiC is more than just increasing the redundancy of the multiple barrier concept. Moderate wall thicknesses provide long breakthrough times and small source terms already. Additional potting with a special compound results in a further upgrading of the canister properties. Therefore a new quality of long-term stable immobilization can be achieved. The available know-how and the technical-technological state-of-the-art meet the requirements for the industrial production of SSiC canisters for the different HLW types.

The SSiC encapsulation as supplementary retention barrier offers new options for repository sites since the host rock alone cannot guarantee the safety requirements.

Acknowledgements

In the recent years the above mentioned proposals were submitted to the relevant

Governmental institutions on Federal and State level in Germany. The authors contacted also decision makers in the nuclear community and persons, who participate in public discussions about nuclear matters with claimed expertise. In most cases there was zero response.

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