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HIGHLIGHTS OF NUCLEAR CHEMISTRY 1995

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Abstract

In this report 9 topics of the work of the Nuclear Chemistry group in 1995 are highlighted. A list of publications and an overview of the international cooperation is given.

19 refs., 19 figs., 2 tabs., 2 app.

Keywords

nuclear chemistry

thermochemical properties

transmutation

Molten Core Concrete interaction

heat conduction

fusion blankets

thorium-based fuel

CONTENTS

1. Introduction	5
2. The actinide laboratory	7
3. Transmutation of technetium	9
4. The chemical form of cesium in HTR fuel	11
5. Release of fission product tellurium during nuclear reactor accidents	13
6. Detailed study of fission product behaviour during Molten Core-Concrete interactions	15
7. Physico-chemical properties of thorium-based fuel	19
8. EXOTIC-7	22
9. Thermal diffusivity and thermal conductivity measurements	26
10. A contribution to lamp development: thermochemical properties of dysprosium aluminates	28
REFERENCES	29
APPENDIX A. List of publications Nuclear Chemistry 1995	31
APPENDIX B. International cooperation	34

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1. Introduction

This report is the second of a series "Highlights of Nuclear Chemistry". Again, the topics are chosen such as to give a representative impression of the chemical research in the business unit Nuclear Energy.

E.H.P. Cordfunke

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2. The actinide laboratory

In 1990 ECN has started a research programme on the partitioning and transmutation (P&T) of long-lived radionuclides that are present in the wastes arising from nuclear power generation, the so-called RAS programme. The goal of this programme is to achieve a substantial reduction of the radiotoxicity of the nuclear waste by means of nuclear reactions such as fission or capture. In the first years this programme was focussed on the reactor physics aspects to investigate whether specific radionuclides (actinides and the fission products iodine and technetium) can be transmuted, which reactor types are to be used for this purpose, and which scenario's are most effective [1].

Next the question of the fuel/target requirements was addressed: what are the most likely compounds to be used and how to fabricate them as pure phases or as mixtures. This is studied by a series of irradiation tests in the High Flux Reactor (HFR) at Petten, which are performed within the frame of the EFTTRA cooperation [2, 3], and by laboratory studies of the fabrication and the properties of specific compounds. Emphasis is laid on the development of non-fertile fuel, consisting of a mixture of the radionuclide to be transmuted and a neutron-inert material (e.g. $MgAl_2O_4$, CeO_2 , ZrN), the so-called inert matrix.

Recently, the problem of separation of radionuclides from waste streams, specifically americium, has also become part of the programme [4]. Due to its similarities with the lanthanides (a major group of fission products), the separation of Am from PUREX waste stream by traditional liquid-liquid extraction is very difficult. Therefore, investigations of the extraction of americium by the Selective Liquid Membrane (SLM) technique have been started.

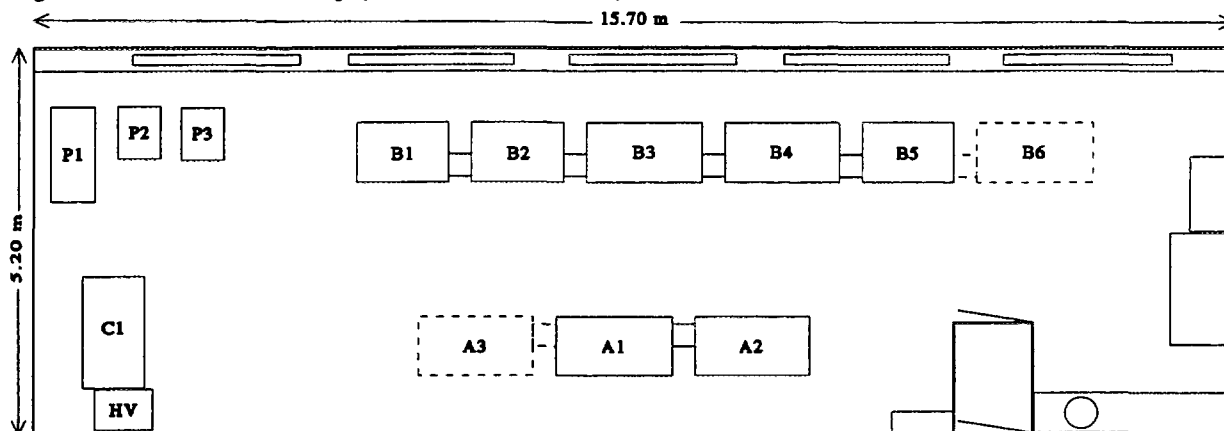
For the work on separation of actinides and the fabrication of fuels, facilities are required to handle reasonable quantities of radioactive materials such as the alpha-emitting actinides and beta-emitting fission products like technetium. Since such facilities were not available at the Petten site, the design of a special actinide laboratory was started in 1994. A space of about 16x6 m at the north side of the Hot Cell Laboratory (HCL) building was selected for this laboratory. In the course of 1995 the infrastructure was adapted according to the defence in depth philosophy (see below): the existing offices were reconstructed into a laboratory environment, the ventilation system was changed to obtain an underpressure with respect to the surrounding rooms, special filters were installed in the off-gas system.

The facilities of the actinide laboratory are located in eight glove boxes, which were fabricated by Mecaplex AG. The glove boxes are made of stainless steel and contain layered reinforced glass windows in the front and rear side, which are equipped with glove ports containing butyl/neoprene gloves. The boxes are operated at under pressure with respect to the laboratory environment; the discharged gases are thoroughly filtered before being supplied to the central off-gas system of the HCL.

The glove boxes are installed in three independent lines (Figure 2.1):

- Line A consists of two boxes interconnected by an antechamber. They are flushed with air and contain connections for water supply and drain off. In these boxes aqueous actinide solutions can be handled, as is necessary for the SLM extraction facility.
- Line B consists of five boxes which are also interconnected by antechambers.

Figure 2.1 Schematic drawing of the actinide laboratory.



These boxes are flushed with argon, which is purified to remove oxygen, nitrogen, carbon dioxide and water (residual values < 1ppm O₂ and H₂O). In this purified atmosphere oxygen sensitive compounds such as nitrides can be handled safely. Facilities that will be installed in the boxes include several high-temperature furnaces (up to 1600 °C) for preparation of targets for irradiation experiments, and thermal analysis (DSC, TGA), light microscopy and EMF equipment for characterisation of materials.

- Line C consist of a single box which is equipped with a high-temperature X-ray powder diffraction facility. The box is flushed with nitrogen.

Each line is equipped with a Syntax posting port to allow easy transfer of material between the different lines. The facilities will become operational in the course of 1996, after a period of thorough testing with non-active materials. Extension of the number of glove boxes is foreseen.

R.J.M. Konings

3. Transmutation of technetium

The fission products technetium (Tc-99, half-life $2.13 \cdot 10^5$ year) is one of the long-lived nuclides in high-level waste that dominate the beta radiotoxicity for more than a million years. Although after some centuries the beta toxicity will remain far below the alpha toxicity of the actinides, technetium and iodine contribute significantly to the risk-during-storage in those scenarios where leaching and transport by water are considered. This is due to their high mobility in these geological environments as a result of which they can migrate easily away from underground repositories and, hence, eventually reach the biosphere.

Technetium can be transmuted by single neutron capture into stable ruthenium-100. However, serious problems are encountered in the technical realisation. Especially the selection of suitable target materials is far from trivial since the requirements with respect to fabrication, stability and compatibility are high because of the necessary long irradiation time (high neutron fluence).

To investigate the irradiation behaviour of technetium metal, three samples were irradiated in the High Flux Reactor at Petten in 1994. This irradiation has been followed by post-irradiation examinations of which the first results are being reported here. The work was performed in the frame of the EFTTRA-collaboration [5].

Technetium as well as ruthenium have a hexagonal crystal structure and they form a solid solution from 0 to 100 %. Although the volume of the solid solution decreases with increasing Ru-content, anisotropic swelling due to neutron irradiation can be expected for this type of crystals. Therefore measurements of the length and diameter have been made for three irradiated rods (see Table 3.1). For rods A and B a Mitutoyo digimatic micrometer was used, for rod D the diameter has been obtained from micrographic examination of a cross section. Comparing these numbers to the average pre-test measurements, it can be concluded that no significant swelling of the pellet has occurred at the present burn-up. This is also confirmed by optical microscopy: the microstructure of the technetium after the irradiation does not reveal significant differences with the pretest structure.

Table 3.1 *Results from the diameter and length measurements for the Tc samples*

sample	diameter (mm)		length (mm)	
	pre-test	post-test	pre-test	post-test
A	4.80 ± 0.01	4.83 ± 0.01	25.05	25.09
B	4.81 ± 0.02	4.84 ± 0.01	25.05	25.12
D	4.81 ± 0.02	4.83 ± 0.03		

The ruthenium concentration of the irradiated pellet D has been measured by electron-probe microanalysis (EPMA) of two sections that have been cut at 5 mm from the bottom (D1) and at 5 mm from the top (D2). The results are shown in Figure 3.1. It can be seen that a sharp decrease of the Ru-concentration occurs in the outer $50 \mu\text{m}$ of the pellet after which the concentration levels off to a constant value of about 5.7 % for section D1 and 5.9 % for section D2. This effect is due to the resonance self-shielding in the epithermal energy range. The mean Ru-concentration calculated from the EPMA curves was (6.9 ± 0.4) %. The mean Ru-concentration was also determined by isotope dilution mass spectrometric

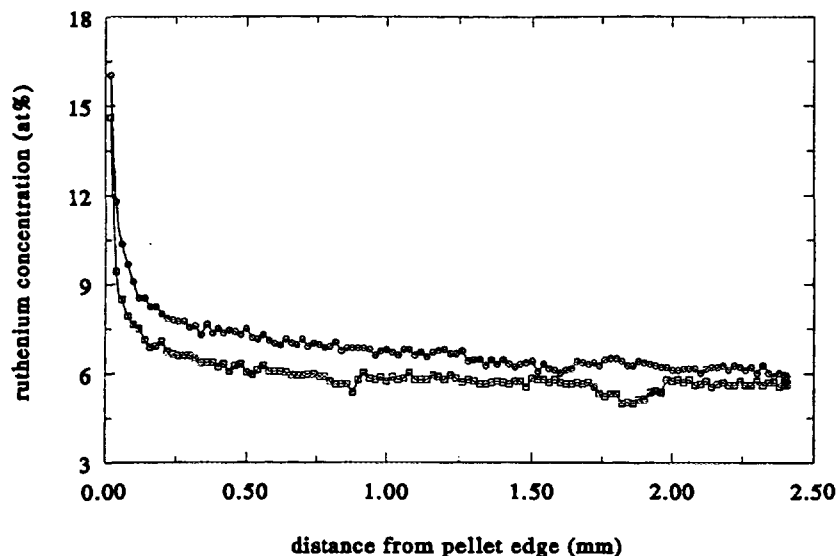


Figure 3.1 *The radial distribution of ruthenium in two sections of the irradiated technetium. \circ , D1; \square , D2.*

(IDMS) analysis of 100 mg samples (disks) of sections D1 and D2, yielding (6.4 ± 0.2) % which is somewhat lower than the result obtained by EPMA.

From the present results it can be concluded that metallic technetium in the form of casted pellets shows no significant swelling under neutron irradiation. However, in the present experiment a relatively low burn-up was reached (~ 6.5 %) and high burn-up studies are required to support this conclusion. Therefore, one of the three technetium samples from the present experiment (containing rod A and B) has been refabricated and will be re-irradiated for a period of at least two years to obtain a burn-up of about > 20 %.

R.J.M. Konings

4. The chemical form of cesium in HTR fuel

The high-temperature reactor is a modular reactor with unique safety features. The operating temperature is 900°C which makes it very well-suited for combined heat and power generation. The reactor is cooled with helium, which is chemically inert and has no objections with respect to the neutronphysics.

The fuel elements are graphite spheres with a diameter of 6 cm. ECN studies a reactor design based on the *Peu-à-Peu* (PAP) concept of KFA Jülich, in which the spheres are brought in the reactor bit-by-bit (*Peu-à-Peu*). Each fuel element is filled with 10.000-15.000 fuel particles, each 1 mm in diameter. The chemical safety of the HTR relies, to a large extent, on the coatings of the fuel particle. A fuel particle consists of a UO₂ fuel kernel, surrounded with several carbon layers and a gastight SiC coating. Tests have proven that irradiated particles can resist temperatures of 1600°C without any significant deterioration of the coating layers.

The fission products are slowed down by the porous carbon layer, which protects the coating for radiation damage. The porous carbon layer acts also as the buffer volume which provides space for the released fission gases.

Cesium is one of the well-known radiotoxic fission products and it is therefore important to know the chemical form of Cs inside the HTR fuel particle. Graphite can bind many metals by sorption, but it can also form so-called intercalation compounds. Intercalation compounds are compounds with a layered structure as depicted in Figure 4.1. The graphite-cesium ratio inside the particle at intermediate values for the burnup (5-10% FIMA) is such that the formation of C₆₀Cs (not to

Figure 4.1 *Schematic drawing of an cesium graphite intercalation compound*

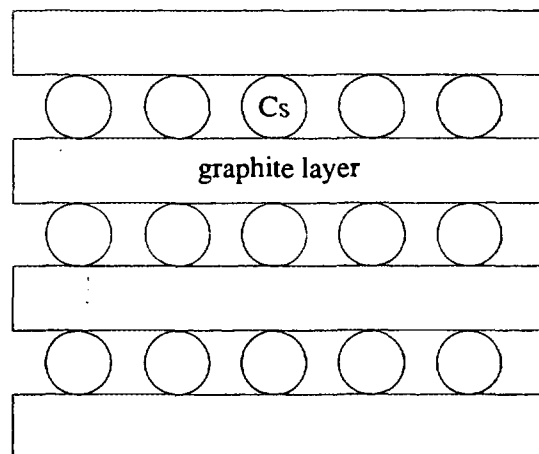
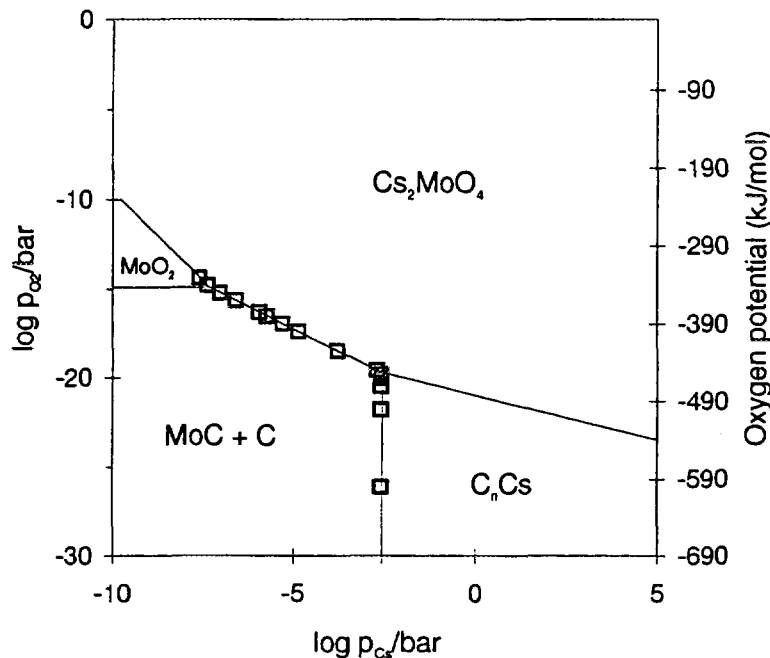


Figure 4.2 Predominance area diagram of the C-Cs-Mo-O system at 1200 K. The boxes refer to the actual cesium and oxygen pressure, calculated for the HTR fuel at 5.5% FIMA.



be confused with the fullerene compounds) is thermodynamically favorable. All cesium graphite compounds (C_nCs) decompose in vacuum above 800 °C. It is not known, however, if these cesium graphite compounds are stable in the confinement of the fuel particle at high temperatures. Therefore cesium graphite compounds (C_{60}Cs , C_{48}Cs and C_8Cs) were synthesized by exposing graphite (or amorphous carbon) to a saturated cesium vapour or a weighed amount of cesium metal at a temperature of 300–400 °C. The cesium-graphite compounds are extremely sensitive to oxygen and have to be treated in argon-filled gloveboxes.

The compounds were brought in a gastight molybdenum cup and were heated to 1700 °C in a Differential Thermal Analysis apparatus. No heat effect indicative for decomposition could be measured.

The chemical form of cesium was calculated for the HTR fuel particle thermodynamic conditions [6]. A so-called predominance diagram for the C-Cs-Mo-O system can be used to identify the stability areas of Cs-compounds for different Cs and O_2 pressures (Figure 4.2). This diagram shows that at low oxygen potentials (< -450 kJ/mol), the cesium pressure is governed by the $\text{C}/\text{C}_{60}\text{Cs}$ equilibrium. At higher oxygen potentials the Cs_2MoO_4 is formed and lower cesium pressures will be found.

Due to the large amount of cesium in the fuel particles, a cesium pressure of 100 bar can be easily reached under normal circumstances, when no stable cesium compounds would be formed. It seems that in the HTR fuel particle the Cs pressure remains relatively low as a result of the formation of either Cs_2MoO_4 or C_{60}Cs .

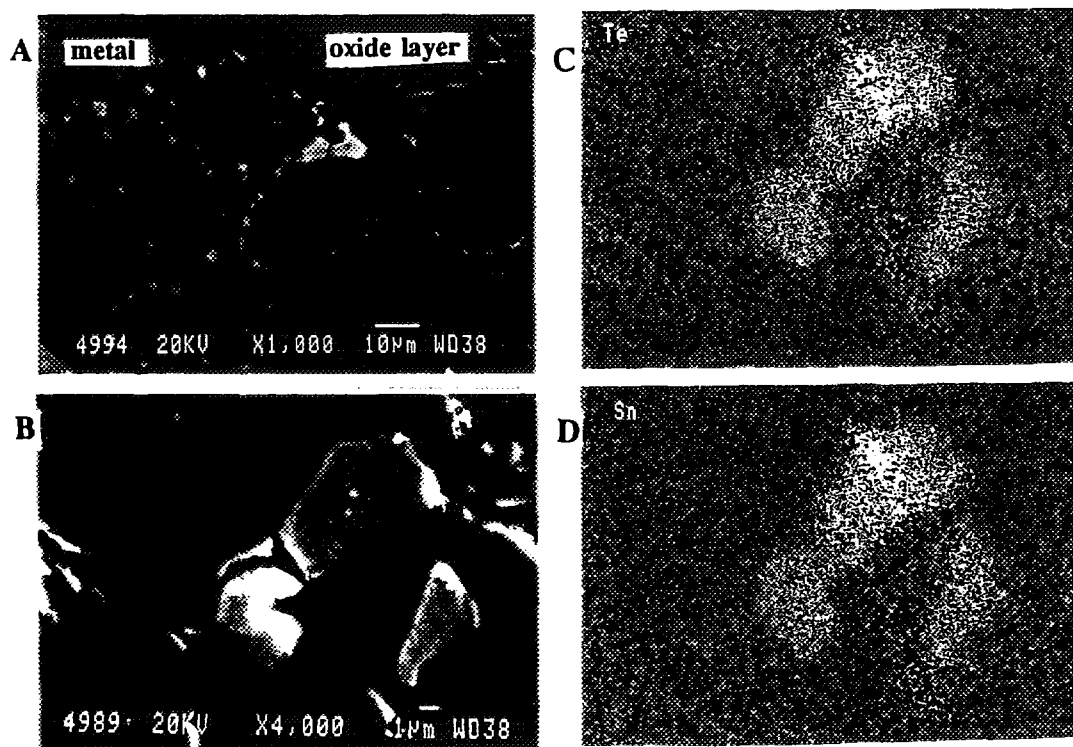
R.P.C. Schram

5. Release of fission product tellurium during nuclear reactor accidents

Tellurium is, as a precursor of the very radiotoxic iodine and due to its volatility, a very important fission product. In the unlikely case of a nuclear reactor accident, tellurium can contribute largely to the health- and environmental consequences; release of tellurium partly determines the release of iodine, and tellurium can, as a vapour, easily be transported from the reactor core through the primary cooling system into the containment building. However, the chemical state of tellurium is of key importance to its release behaviour. During a reactor accident, interaction of tellurium with other fission products, and with reactor construction materials, alters the chemical state of tellurium, and consequently alters its release behaviour. Knowledge of these interactions, and of the chemical state of tellurium during normal and off-normal reactor conditions is necessary for reliable calculations of the release tellurium.

During normal reactor conditions tellurium is present in the uranium oxide fuel matrix in its elemental form or as a cesium telluride. In the initial stage of a reactor accident the fuel temperature rises, and volatile fission products are transported from the fuel into the gap between the fuel and its cladding. Tellurium then reacts with the inner surface of the zirconium based cladding and stable, non-volatile zirconium telluride compounds are formed. This chemical interaction of tellurium with the Zircaloy (1.5%Sn, 0.2%Fe, 0.1%Cr, Zr balance) cladding will lead to a strong retention of tellurium in the reactor. When the temperature of the fuel and the cladding rises further, rapid reaction of the outer surface of the Zircaloy cladding with steam will occur, leading to formation of zirconium oxide

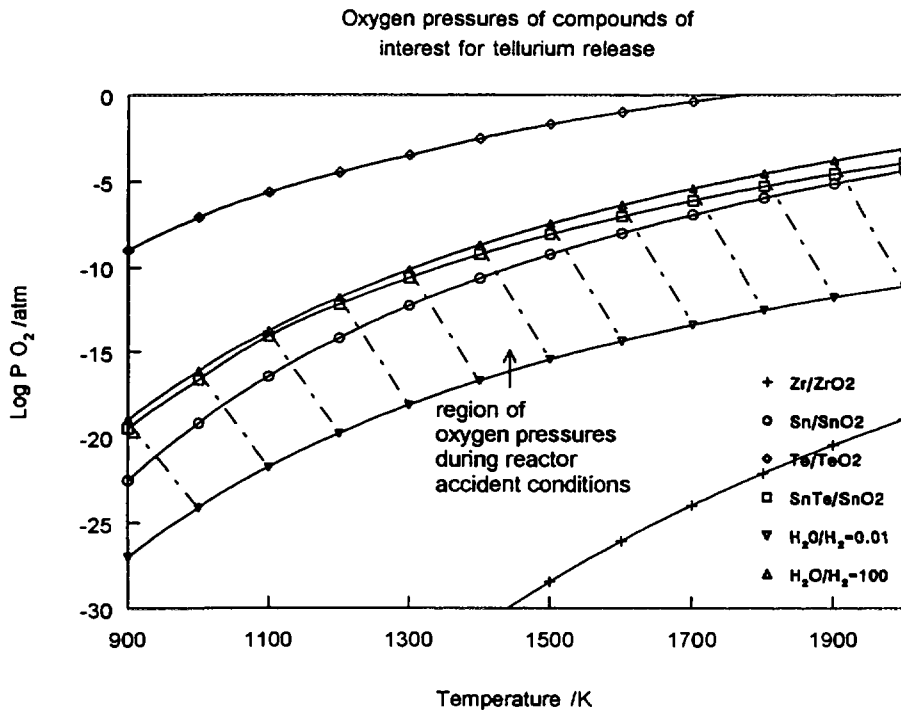
Figure 5.1 A:SEM picture of metal/oxide interface after reaction of Zircaloy with tellurium and with oxygen. B:SEM picture of light inclusions in the oxide layer near the metal/oxide interface.C,D: Te and Sn distribution image of figure B.



and hydrogen gas. This process will finally alter the chemical state of tellurium, and when all zirconium is oxidised, tellurium will be re-evaporated.

This process of Zircaloy oxidation and release of tellurium has been investigated in our laboratory. First, pieces of Zircaloy were heated at different temperatures in an oxygen containing atmosphere to study the behaviour of the cladding additives, tin, iron and chromium, during the oxidation process. Analysis of the cross-sections of the oxidised Zircaloy showed clearly that tin particles are segregated in a narrow rim in the oxide layer. When the thickness of the oxide layer increases, these particles of tin move towards the inner cladding surface, where reaction with tellurium leads to formation of tin telluride, which is shown in the SEM/EDX pictures in Figure 5.1.

Figure 5.2



Release of tellurium from the Zircaloy cladding now is strongly dependent on the local oxygen potential. During a reactor accident the oxygen potential in the reactor is dominated by the ratio of the amounts of steam and hydrogen. Hydrogen is present due to the reaction of steam with Zircaloy. To study the influence of this oxygen potential, experiments were done in which a mixture of zirconium, tin and zirconium-telluride were heated in an atmosphere with different amounts of steam and hydrogen. These experiments showed that at low oxygen potentials tellurium was released from the mixture as tin telluride and at high oxygen potentials, $H_2O/H_2=100$, tellurium was released in its elemental form, due to reaction of tin telluride with oxygen, forming tin oxide and tellurium. This behaviour is supported by chemical equilibrium calculations of the oxygen potentials of the compounds and elements which are of importance in the release of tellurium, as is shown in Figure 5.2. As the vapour pressure of SnTe is lower as that of elemental tellurium, the formation of SnTe results in a lower release rate of tellurium.

Robert de Boer

6. Detailed study of fission product behaviour during Molten Core-Concrete interactions

The nuclear industry traditionally has great interest in thermodynamic properties to calculate the chemical processes occurring in, for instance, nuclear power plants. The need to calculate the release behaviour of fission products and their compounds during off-normal operating conditions is one of the major topics in reactor safety analysis. The use of chemical thermodynamics has proven to be the tool to achieve this, especially under those conditions where reliable unequivocal experimental data is hard to obtain.

The thesis of Mark Huntelaar: "Thermochemical Properties of some Alkaline Earth Silicates and Zirconates - fission product behaviour during Molten Core Concrete Interactions" [7] tries to make a contribution to a better understanding of the chemical processes occurring during an ex-vessel Molten Core Concrete Interactions (MCCI) accident with a western-type of nuclear reactor. The emphasis in the thesis is on the radiological hazardous fission product barium oxide and strontium oxide, since they represent the long-term radioactive contamination and the biological hazard they may inflict to people who come in contact with these compounds. The choice for silica and zirconia is also a very simple one: SiO_2 is one of the major constituents of concrete and zirconia will be abundantly present during severe accidents as a result of the oxidation of the Zircaloy clad which originally contained the nuclear fuel. Consequently, both matrix compounds are responsible for a drastic change in the chemical behaviour of the fission products. This is not only due to the chemical stability of the silicates and the zirconates but also a result of the non-ideal chemical behaviour of mixtures containing the matrix compounds.

After a short introduction in the history of research in nuclear safety followed by the state-of-the-art on molten core concrete interactions the thermodynamic stabilities of the relevant barium and strontium silicates and zirconates are determined. The entropy and low-temperature heat-capacity of several compounds are measured by adiabatic calorimetry from 5 to 325 K. Whereas the high-temperature heat capacity is obtained from drop-calorimetric measurements. The standard molar enthalpy of formation has been determined with isoperibol solution calorimetry. For convenience a list of all measurements is given in Table 6.1.

Using the thermodynamic properties obtained in the previous chapters phase diagram calculations have been performed in the ternary system $\text{SrO-SiO}_2\text{-ZrO}_2$. Where necessary DTA-measurements have been performed to obtain additional phase diagram information such as melting points, liquidus and solidus temperatures and enthalpies of fusion. Although it was originally intended to optimize the binary systems SrO-SiO_2 , $\text{SiO}_2\text{-ZrO}_2$ and SrO-ZrO_2 with different thermochemical models, only the Redlich-Kister model yielded acceptable results. The Associate model as well as the Quasi-Chemical model both failed to describe the SrO-SiO_2 sufficiently. Finally a ternary system was calculated using the optimized excess parameters obtained for the binary systems.

Calculations in the binary and ternary systems confirm the expected suppression of the chemical activity of strontium oxide in the liquid. Validation calculations on a ten-component system, using a dedicated thermochemical database of ECN and AEA-Technology which included excess interaction parameters, performed

Table 6.1 A list of the thermodynamic properties determined in the present study.

compound	$S^\circ(298.15)$	$\{H^\circ(T)-H^\circ(298.15)\}$	$\Delta_f H^\circ(298.15)$
<i>solid</i>			
BaSiO ₃		x	x
BaZrO ₃	x	x	x
α -SrSiO ₃		x	x
am-SrSiO ₃		x	x
δ -SrSiO ₃			x
Sr ₂ SiO ₄	x	x	x
SrZrO ₃		x	x
SrZrSi ₂ O ₃	x	x	x
Sr ₇ ZrSi ₆ O ₂₁	x	x	x
ZrSiO ₄	assessment	assessment	assessment
<i>gas</i>			
BaO			assessment
SrO			assessment

for the Third-Framework Programme of the European Union show similar results. In these calculations the release of SrO, BaO, CeO₂ and La₂O₃ have been calculated as a function of fuel vs concrete concentration. Besides this two different types of concrete have been simulated: SiO₂-rich European siliceous concrete and CaO-rich American limestone concrete. The results from the calculations clearly indicate a dependency with the SiO₂ content in the concrete-fuel debris (Figures 6.1 and 6.2), so that can be concluded that SiO₂ (and ZrO₂) is the key-factor determining the release of strontium oxide. Unexpectedly, however, the same calculations indicate that the chemical activity of strontium oxide is enhanced at low

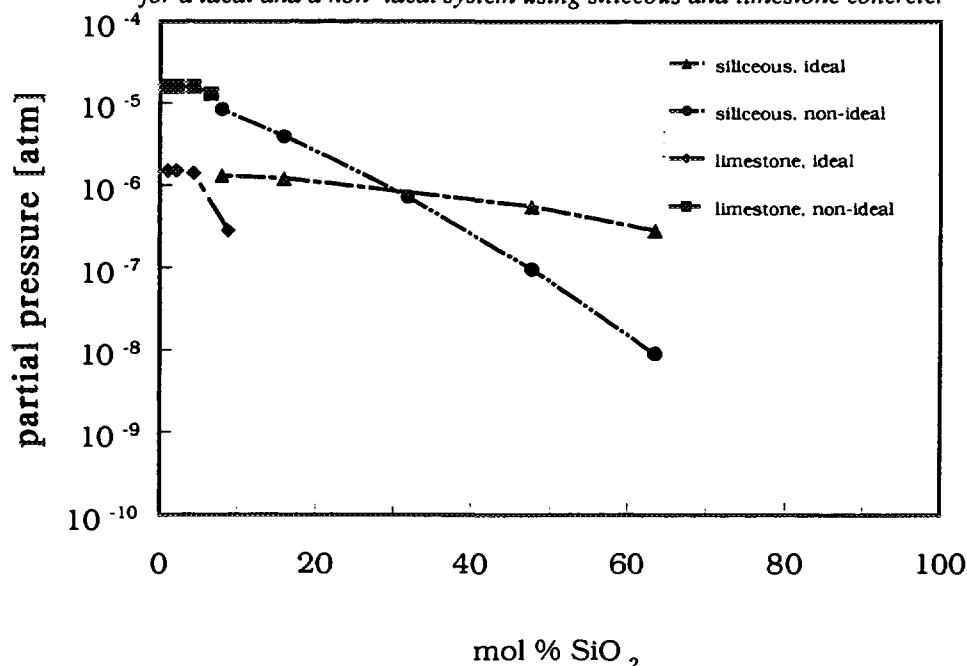
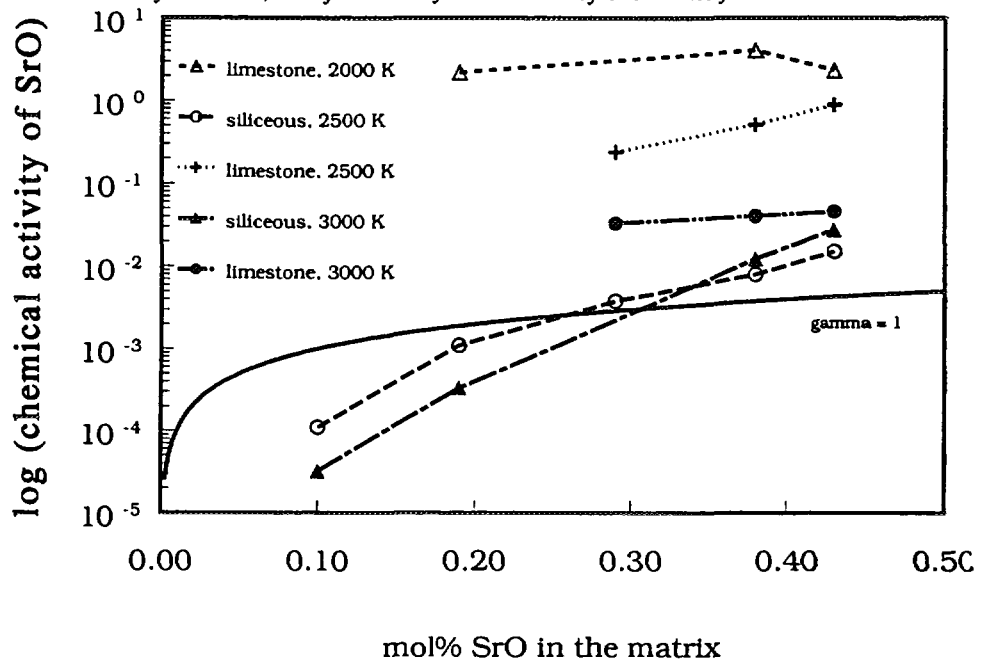
 Figure 6.1 The partial pressure of SrO(g) at 3000 K as a function of the amount of SiO₂ for a ideal and a non-ideal system using siliceous and limestone concrete.


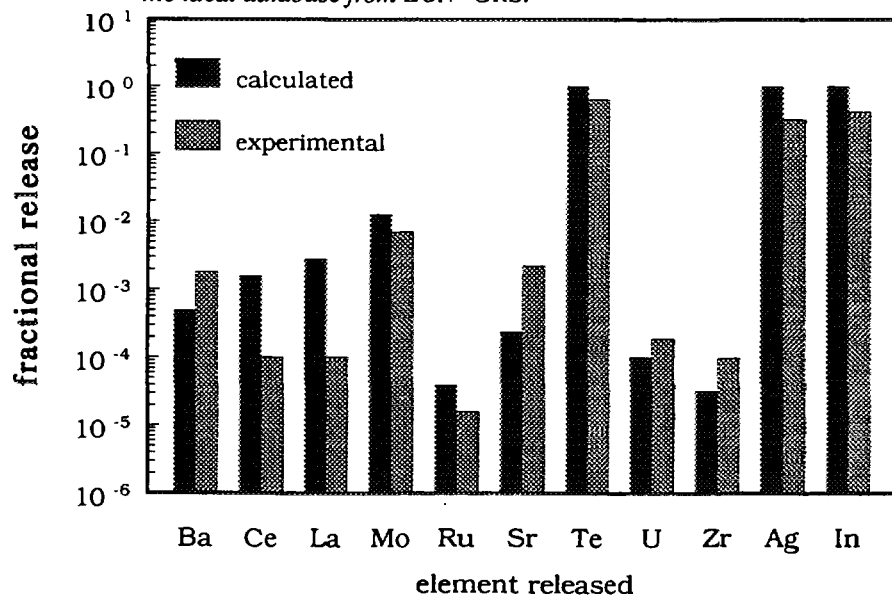
Figure 6.2 The chemical activity of SrO at different temperatures and for the two types of concrete, as a function of the amount of SrO in the fuel-concrete matrix.



SiO₂ concentrations. This is caused by high contents of CaO in these mixtures.

This result explains why in the ACE-L6 and L8 validation calculations, which also have been performed within the Third Frame Work, using an idealized thermochemical database from ECN and GRS (Gesellschaft für ReaktorSicherheit und anlagen), the ACE-L6 experiment (Fig. 6.3) is much better calculated than the L8. Due to the limestone-concrete used in the L8 simulation experiment fission product release is enhanced and consequently cannot be predicted using an ideal database. Whereas due to the siliceous concrete used in the L6 experiment at

Figure 6.3 The results from the validation calculations for the ACE-L6 experiment using the ideal database from ECN-GRS.



early stages in the accident the release is enhanced and is depressed later when more SiO_2 becomes involved due to ablation of the concrete. The net effect is that the overall chemical activity becomes almost ideal and that a simple ideal database is sufficient to calculate the release behaviour.

M.E. Huntelaar

7. Physico-chemical properties of thorium-based fuel

At present, most nuclear power stations are fuelled with uranium, where ^{235}U is the fissile material and ^{238}U acts as the matrix. The presence of ^{238}U in the fuel leads to an enhanced production of transuranium elements with high radiotoxicity, such as americium and curium. The use of ^{238}Th in nuclear fuel has the advantage of a relatively low actinide production. Since ^{238}Th is not fissile, some startup material such as ^{235}U has to be added to the fuel. We will consider thorium-based fuel that consists of ^{238}Th and ^{235}U , in the chemical form of a solid mixture of UO_2 and ThO_2 .

The performance of nuclear fuel is highly dependent on physico-chemical properties such as the thermal conductivity, the oxygen potential and the Gibbs energy (chemical stability). Of course, these properties should be investigated for different reactor operating conditions, i.e. for different values of the burnup and the temperature. But before the dependence of the burnup is investigated, first the properties of the fresh unirradiated fuel should be known. Therefore, a literature study and an assessment of the thermal properties of the system $\text{UO}_2\text{-ThO}_2$ was initiated by the Nuclear Chemistry group of ECN [8,9].

One important aspect of the fuel is the chemical stability at elevated temperatures. The components of the fuel should form a stable solid solutions with preferably no phase transitions in the working temperature range. Thorium oxide and uranium oxide have much higher melting temperatures than most of the fission product compounds that may be formed. In Figure 7.1 a plot of the phase diagram of the mixture is shown. Above 3640 K the mixture is completely liquid and below 3120 K the mixture is completely solid. The phase diagram shows that the mixture is

Figure 7.1 Phase diagram of the $\text{UO}_2\text{-ThO}_2$ system. The filled symbols refer to solidus points, the open symbols refer to liquidus points.

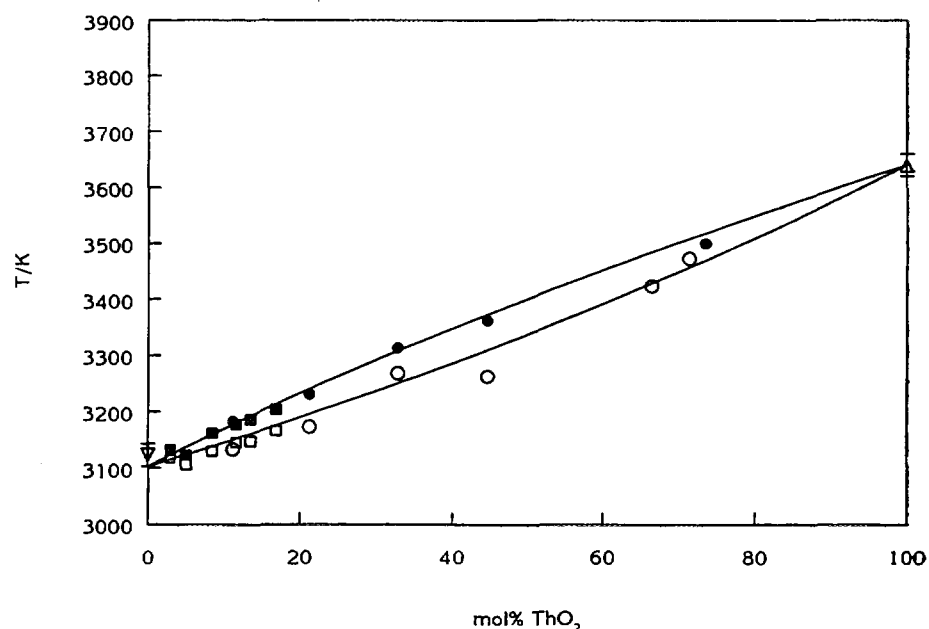
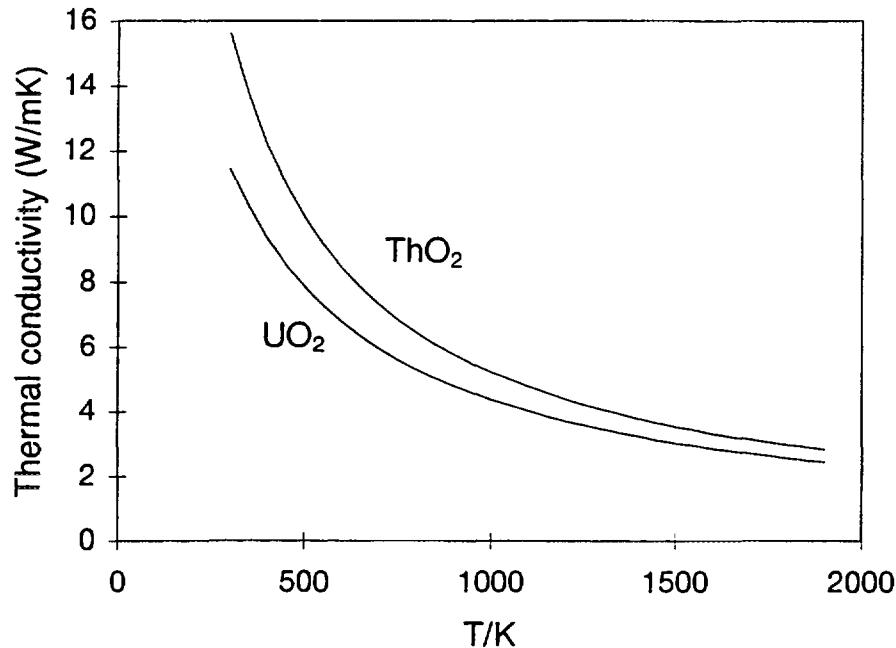


Figure 7.2 The thermal conductivity of UO_2 and ThO_2 .

behaves ideal, which is supported by measurements of the lattice constant. UO_2 and ThO_2 both have the fluorite crystal structure and form a continuous solid solution. The UO_2 - ThO_2 system has a good chemical and thermal stability.

The thermal conductivity of the fuel is one of the key parameters for optimal fuel performance. The heat transfer from the fuel to the coolant, as well as the average fuel temperature, is dependent on the intrinsic thermal conductivity of the fuel. Poor thermal conductivity may lead to large temperature gradients (resulting in large stress gradients) and undesirable high temperature in the fuel pin. The thermal conductivity of $Th_{1-y}U_yO_2$ (Figure 7.2):

- decreases with increasing temperature
- increases with increasing thorium concentration

The use of thorium has therefore a positive effect on the thermal conductivity of the fuel.

The oxygen potential of the fuel determines the availability of gaseous oxygen in the fuel pin, which in turn, influences the chemical form of the fission products. A large amount of experimental data of the oxygen potential of $Th_{1-y}U_yO_{2+x}$ was retrieved from literature and stored in a database [10]. It was found that the oxygen potential of $Th_{1-y}U_yO_2$:

- increases with increasing temperature
- increases with the excess oxygen concentration (x in $Th_{1-y}U_yO_{2+x}$)
- increases with increasing thorium concentration.

The use of thorium in oxide nuclear fuel leads to a higher oxygen potential and hence a stronger oxidizing fuel than plain uranium oxide. The effect of adding thorium oxide to uranium oxide is illustrated in Figure 7.3.

R.P.C. Schram

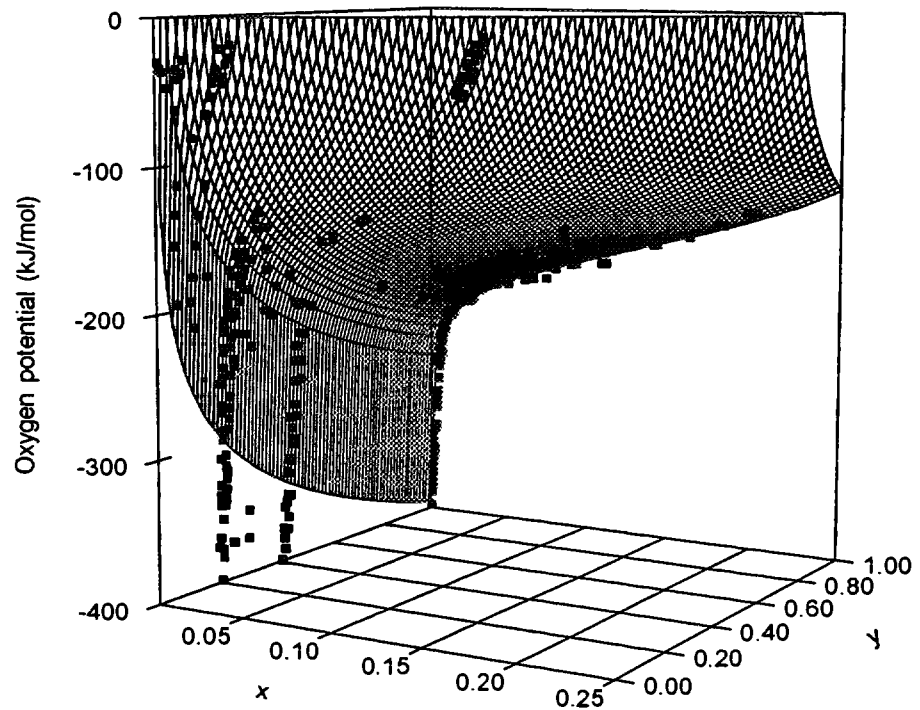


Figure 7.3 Oxygen potential of $U_yTh_{1-y}O_{2+x}$ versus the excess oxygen concentration x and the uranium concentration y ($T=1000-1473$ K)

8. EXOTIC-7

Lithium-containing ceramics are considered as tritium breeding material for fusion reactor blankets. The EXOTIC (Extraction Of Tritium In Ceramics) programme at ECN Petten aims at the development of production routes of lithium ceramics, and optimization of their properties and characteristics with respect to the in-situ release of tritium and material stability, in particular, up to high lithium burnup. This work is carried out within the frame of the European Fusion Technology Programme, in close cooperation with JRC-IAM Petten and jointly with FZK Karlsruhe, ENEA Casaccia, CEA Saclay and AECL Chalk River. Irradiation experiments are performed using the High Flux Reactor (HFR) at Petten to determine the tritium release characteristics of various ceramic breeder materials at different irradiation conditions, like temperature, purge gas composition and burnup. A large series of experiments which are known as EXOTIC-1 to EXOTIC-6 have been performed in the period 1985-93 [11-14].

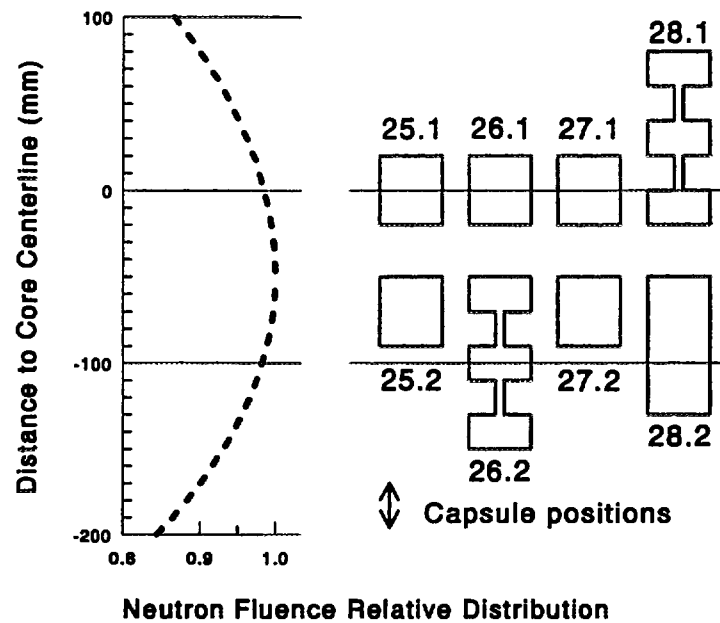
The objective of the EXOTIC-7 experiments performed in 1994/5 has been: to irradiate candidate ceramic breeder materials in the HFR to a high lithium burnup (target $\approx 10\%$) and to determine the effects on the mechanical integrity of pellets and pebble-bed configurations and the effects on tritium-inventory and -release characteristics. The tested breeder materials were candidates within the European Blanket Development Programme [15] and shaped as pellets for Li_2ZrO_3 , Li_8ZrO_6 and LiAlO_2 and as pebbles for Li_2ZrO_3 and Li_4SiO_4 . Two capsules contained a mixture of Li_4SiO_4 and beryllium pebbles. To obtain a high lithium-burnup within a reasonable irradiation time the target materials were enriched with ^6Li to about 50%.

The irradiation of EXOTIC-7 was performed in a TETRA rig, providing four independent parallel irradiation channels in one core position, each channel containing two capsules. The eight capsules were simultaneously operated under independent temperature and reference purge gas conditions [16, 17]. The instrumentation included thermocouples, neutron fluence detector sets and gamma scan wires. Temperature control was performed by gas mixture technique and/or by electrical heaters. Temperature transients have been performed during irradiation to determine tritium residence times.

The irradiation of EXOTIC-7 was started in February 1994 and completed on February 6, 1995. The experiment was placed in position H6 of the HFR for 11 reactor cycles achieving 261 Full Power Days. The position average neutron fluences are about $2.7 \times 10^{25} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$) and $1.3 \times 10^{25} \text{ n/m}^2$ ($E > 1 \text{ MeV}$) [18]. The positions of the capsules relative to the neutron fluence distribution is visualized in Figure 8. The total lithium-burnup achieved ranges between 6% for the Li_4SiO_4 pebbles and 18% for the capsule containing Li_4SiO_4 mixed with Be, see Figure 8. The much higher burnup of the Li_4SiO_4 in the mixed bed with beryllium is due to the lower smear density of the Li_4SiO_4 which results in a lower depression of the neutron flux density. The (n,α) power varied from a range of 24 - 145 W/g at the irradiation start to 22 - 107 W/g at the very end. The amount of tritium produced per capsule varied from 4 to 12 TBq.

Neutron radiography of the rig has been done at several radial positions after the last reactor cycle. No evidence was found for irregularities, i.e. pellet stacks, pebble-beds and claddings were considered essentially intact. Subsequently, dis-

Figure 8.1 Schematic positions of EXOTIC-7 capsules and relative neutron fluence distribution



mantling of the EXOTIC-7 sample holders in the ECN Hot Cell Laboratories started one month after irradiation. The external gamma-scan wires (GSW) and neutron monitors were easily obtained. The unloading of sample materials from the capsules was done in a lead-shielded cell, equipped for handling tritium containing materials.

The pellet containing capsules have been cut and pellets were unloaded as far as possible one by one. The pellet appearance was inspected prior to unloading, but most of the pellets fell apart in fragments after unloading. Three out of five

Figure 8.2 Lithium burnup achieved during 11 HFR cycles (261 Full Power Days) for the various sample materials, calculations based on 50% initial ^6Li -abundance

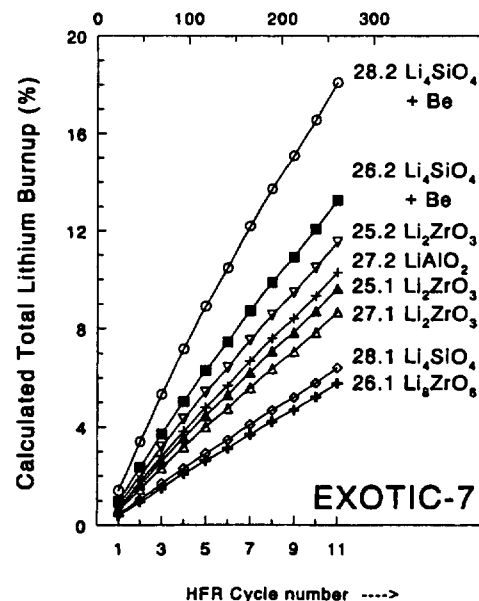


Figure 8.3 SEM picture of the top end of capsule 28.2 after unloading, showing resin impregnated pebbles: large beryllium pebbles and the mixture of small lithium-orthosilicate and small beryllium pebbles

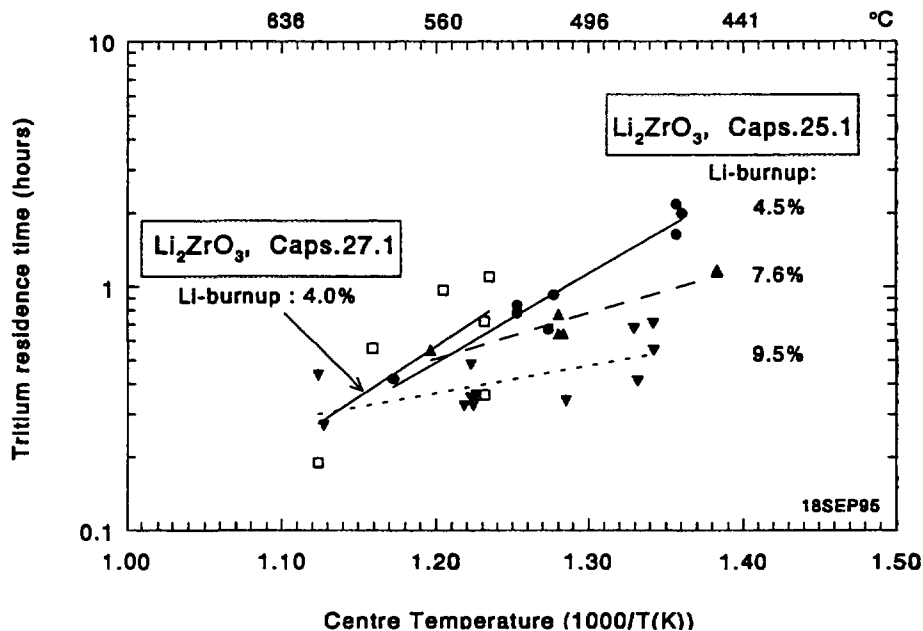


Li_2ZrO_3 pellets in capsule 25.1 remained intact as well as one pellet from 27.1, which had a height of 2.6 mm only. The diameter and height of the pellets and the larger fragments were measured. The dimensional changes were almost in the range of the measurement accuracy (± 0.01 mm).

For unloading the capsules containing pebble-beds a special procedure was followed: in order to obtain information on the undisturbed packed bed structure it was tried to 'freeze' the pebble configuration. Unloading of the pebbles from the beryllium containing capsules (26.2 and 28.2) was very difficult: the mixed-bed pebbles were very 'sticky'. In addition significant amounts of tritium, of the order of 2-20 GBq, were released during unloading. Figure 8 shows a SEM picture of the interior of the top end of capsule 28.2 after unloading and cutting. The pebble masses were visually inspected and small numbers were examined by SEM at moderate magnifications (about 700 x). The Li_2ZrO_3 pebbles (25.2) showed no fracture or cracking. A small number of the Li_4SiO_4 pebbles (28.1) were fractured; SEM results showed the appearance of surface cracks. A slightly higher degree of cracking occurred for the sections irradiated at higher temperatures. Due to the pronounced inhomogeneity of the as-manufactured pebbles it is difficult to draw firm conclusions yet from observing a small batch of irradiated material. The observations presented here will be followed by more detailed microstructural investigations. This applies in particular to the possible dimensional changes of the irradiated mixed beds with beryllium pebbles.

A number of temperature transients have been performed during irradiation of EXOTIC-7 in order to monitor the tritium release characteristics. So far only a few transients during the reactor cycles 5, 9 and 11 have been analysed by fitting the measured tritium release rate during the transients with the diffusion model DIFFIT as was also used for analysis of residence times in EXOTIC-6 [11, 12, 14]. In most cases proper fits were obtained. Some results for tritium residence times of Li_2ZrO_3 from capsules 25.1 and 27.1 are shown in Figure 8. Note that the residence times are given as function of the maximum measured temperature,

Figure 8.4 Tritium residence times of Li_2ZrO_3 pellets irradiated in EXOTIC-7 capsules 25.1 and 27.1 at the indicated lithium burnup. The temperatures are the maximum values at the central axis of the specimens



being the average of the three central thermocouples. At a later stage the results will be coupled to the volume averaged temperatures. As can be observed from Figure 8 the residence times of Li_2ZrO_3 -CEA-EV is almost equal to that of Li_2ZrO_3 -CEA at a similar burnup. However, the residence time of Li_2ZrO_3 -CEA (25.1) at 7.6% lithium burnup is slightly lower compared to that at 4.5% burnup for centre temperatures < 500 °C and dropped even more during the last cycle with the lithium burnup of 8.7-9.6 %. Based on these data it is concluded that the tritium release rate of the Li_2ZrO_3 pellets for temperatures > 500 °C is not influenced by the burnup up to about 9%. However, at centre temperatures < 500 °C a decrease of the tritium residence time is observed starting after a lithium burnup of about 5%.

Conclusions The EXOTIC-7 experiment, which was ended in february 1995 has been successful in achieving a high lithium burnup during 11 HFR cycles (261 Full Power days). The calculated total lithium burnup for the eight capsules is in the range of 6-18%.

The pellet-stacks and pebble-beds have remained essentially intact. For one capsule three out of five Li_2ZrO_3 pellets were found intact after unloading.

The tritium residence time of Li_2ZrO_3 pellets at higher temperatures has not changed during the irradiation. After a lithium burnup of about 5% the residence time at lower temperatures shows significant decrease with increasing burnup.

The programme will continue with further analysis of tritium release characteristics, inventory, lithium burnup and microstructure. Analysis of in-situ temperature transients will be extended, including the application of a desorption model. On completion of the detailed analysis of dosimetry results, the correct fluences, lithium burnup and temperature fields will be calculated.

J. van der Laan

9. Thermal diffusivity and thermal conductivity measurements

At ECN laserflash equipment has been established in order to measure the thermal diffusivity and the thermal conductivity of solid materials (e.g. metals and ceramics) in the temperature range between 20 °C and 1000 °C. At present the equipment is used to study the thermal conductivity of nuclear fuel and of metallic reactor components. The thermal diffusivity of the alloy Zircaloy is shown in Figure 9.1.

With the laserflash method a disk-shaped sample (diameter 8-13 mm, thickness 0.5-4 mm) is heated under vacuum to a constant temperature in a water cooled high temperature furnace. The sample is mounted adiabatically in an aluminumoxide sampleholder. The front side of the sample is heated uniformly by the light pulse of a ruby laser. The ruby laser has a wavelength of 694 nm, a pulse duration of 1 ms and a pulse energy between 8 J and 28 J. The time dependence of the temperature rise on the rear side of the sample is detected with a chromel-alumel thermocouple. The thermocouple signal is recorded and analyzed with an amplifier, a high speed oscilloscope and a computer. The time between the laserpulse and the temperature of the rear side of the sample to reach half of its final value is used to calculate the thermal diffusivity. The total temperature increase of the sample, induced by a laser pulse, is approximately 5 °C. An example of the thermocouple response is shown in Figure 9.2

The accuracy of the equipment was controlled with tungsten, Zircaloy and aluminumoxide samples of various thicknesses. The systematic error was found to be approximately 5%, depending slightly on the material properties. In the case of translucent or non-electrically conducting samples one or both sides of the sample are coated. This technique has been used to study ceramics such as CeO₂ and

Figure 9.1 *The thermal diffusivity of Zircaloy, as measured on two samples with different thicknesses.*

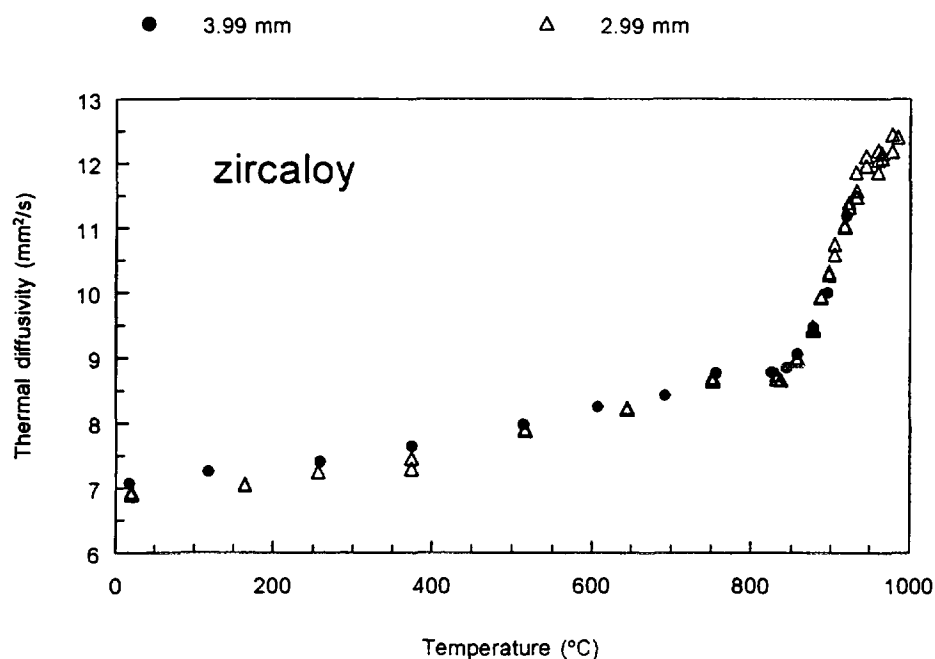
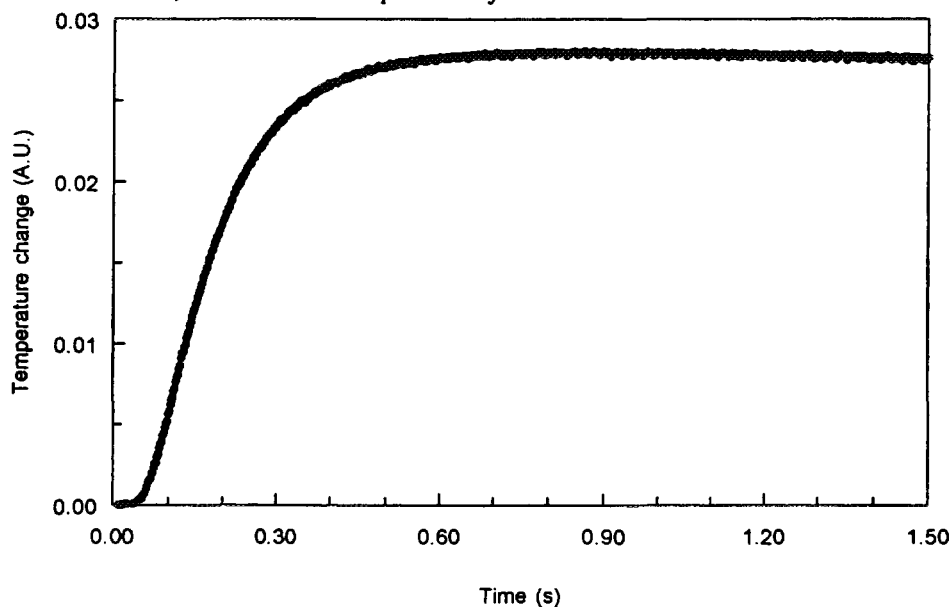


Figure 9.2 *The time dependence of the temperature increase of the rear side of a Zircaloy disk, with an initial temperature of 500 °C.*



CePO₄. The thermal conductivity (λ) can be calculated (Eq. 9.1) combining data on the thermal diffusivity (α), the density (ρ) and the specific heat (C_p). The temperature dependence of the specific heat and the density can also be determined at ECN.

$$\lambda = \alpha \rho C_p \quad (9.1)$$

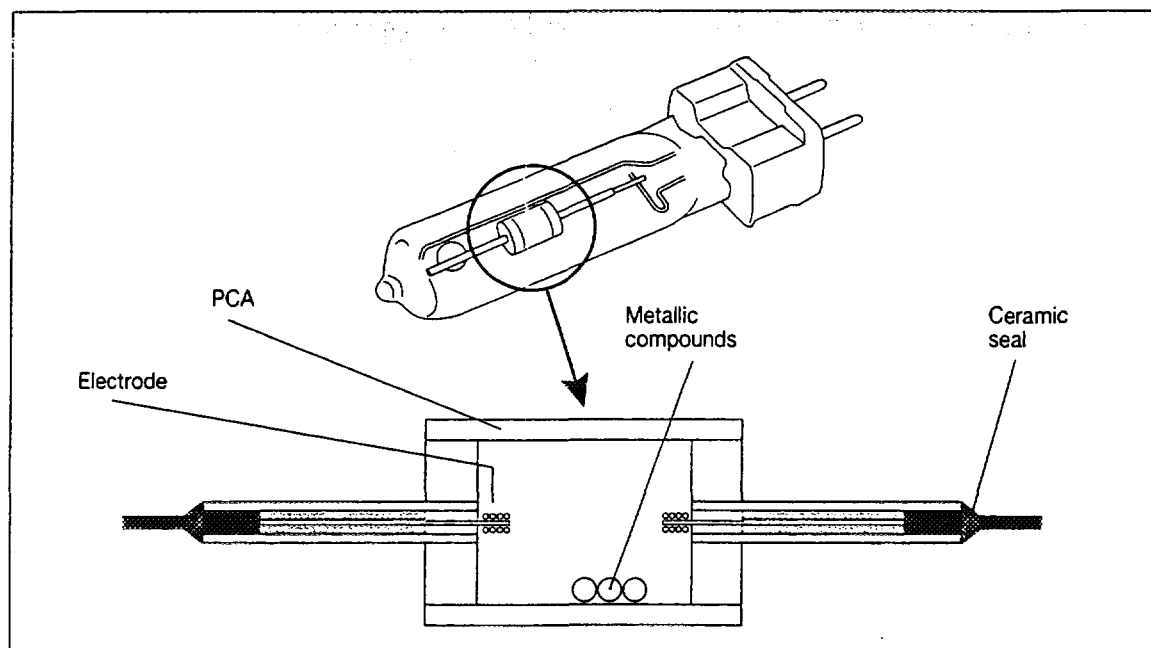
In 1996 the temperature range of measurement will be extended up to approximately 1700 °C using optical temperature detectors. In general, the thermal diffusivity and the thermal conductivity of most materials (e.g. metals and ceramics) can be studied accurately.

K. Bakker

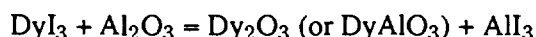
10. A contribution to lamp development: thermochemical properties of dysprosium aluminates

On the request of Philips Lighting, Eindhoven, thermochemical properties of dysprosium aluminates have been measured. These properties are needed to study the reactions that can occur in Mastercolour lamps, a new type of high-pressure lamps which have a high performance and good colour properties by the addition of rare earth halides. Reactions of these halides cause damage to the discharge tube wall, which leads to leakage or rupture of the arc tube, and, consequently, limit the performance.

Figure 10.1 *Structure details of a Mastercolour lamp*



In order to describe the mechanism of the wall reactions, a hypothesis has been formulated in which the liquid metal halides react with the Al_2O_3 -wall to form aluminates, such as DyAlO_3 or $\text{Dy}_3\text{Al}_5\text{O}_{12}$:



As a result, wall material disappears and a crater is formed around the electrode. The AlI_3 , which is formed in this reaction, provides enough acidity to further dissolve Al_2O_3 at these high temperatures ($\sim 1275 \text{ K}$).

In order to calculate the possible reactions, the enthalpies of formation of DyAlO_3 , $\text{Dy}_3\text{Al}_5\text{O}_{12}$, and $\text{Dy}_4\text{Al}_2\text{O}_9$ have been determined by solution calorimetry. In addition, the entropies of these compounds have been measured by low-temperature calorimetry (heat capacity measurements) and the enthalpy increments by drop calorimetry. The results have been combined to derive the Gibbs energies of formation, as a basis for the thermochemical calculations [19].

E.H.P. Cordfunke

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APPENDIX B. International cooperation

Institute	Project/subject
AEA Technology, Harwell Laboratory (UK)	MCCI
AECL Chalk River (Canada)	EXOTIC
CEA, Cadarache (France)	EFTTRA, CAPRA
CEA Saclay (France)	EXOTIC
EdF SEPTEN (France)	EFTTRA
ENEA Casaccia (Italy)	EXOTIC
EU	MCCI
	EXOTIC
	EFTTRA
FZK Karlsruhe (Germany)	EXOTIC
	EFFTRA
FZ Jülich (Germany)	lamp development
GRS (Germany)	MCCI, database development
GTT (Germany)	Database development
Hungarian Academy of Sciences (Hungary)	structural analysis of gases
JAERI (Japan)	High Temperature Reactor
JRC-Institute of Advanced Materials Petten (NL)	EXOTIC
	EFTTRA, RAS
JRC-Institute for Transuranium Elements (Germany)	EFTTRA
NRC (USA)	MCCI
OECD/NEA	RAS
Philips Eindhoven/Aken (Germany)	lamp development
Sandia National Laboratories (USA)	MCCI
University of Oslo (Norway)	structural analysis of gases