SPINEL INERT MATRIX FUEL TESTING AT THE HFR PETTEN

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Abstract

Magnesium aluminate spinel is one of the most investigated inert matrix materials, due to its good physico-chemical stability, resistance against neutron irradiation, and its thermal conductivity comparable with conventional UO_2 fuel. At the HFR in Petten various combinations of actinides and spinel have been tested within the EFTTRA programme, with the object to study the transmutation of americium. To study the incineration of plutonium in spinel in a once through mode, the OTTO irradiation was started, which is still being irradiated at the HFR.

From the various irradiation tests it appears that $MgAl_2O_4$ spinel shows a chemical instability at high temperatures and a large volumetric swelling under the influence of high-energy fission products. These disadvantageous properties put severe restrictions on the use of spinel for the partitioning and transmutation of spent fuel.

Introduction

In order to reduce the environmental impact of radioactive waste, innovative concepts are being developed for the recycling of actinides, which make up most of the radiotoxicity of spent fuel. One of the concepts under investigation is the use of inert matrix fuels, which consist of a fissile phase embedded in an inert, i.e. transparent to neutrons, matrix phase. The latter can be either a metal, such as stainless steel or molybdenum (CERMET) or a ceramic material, such as zirconia or magnesium aluminate spinel (CERCER). $MgAl_2O_4$ spinel is thought to be a good candidate due to its low neutron absorption cross section, its relatively large thermal conductivity (slightly larger than that of UO_2), high melting point and its good compatibility with cladding material and reactor coolant. In addition, spinel is insoluble in nitric acid and is considered as a good storage medium for final disposal.

The EFTTRA[1] collaboration has made an extensive study of the use of spinel as inert matrix material. [1,2] The aim of the joint research was to study the transmutation of i) fission products and ii) americium. At first the transmutation of long-lived fission products ^{129}I [3] and ^{99}Tc [4] was investigated. In a later stage various inert matrix materials were tested for the transmutation of ^{241}Am, of which spinel is the most extensively studied.

The irradiation studies at the HFR Petten focused on the mechanical and chemical stability of inert matrices under neutron irradiation and fission. A series of irradiation has been performed at the HFR to specifically investigate the irradiation damage by i) neutrons, ii) neutrons + fission products and iii) neutrons + fission products and enhanced helium generation. The dedicated tests were performed at various temperatures and with different neutron fluences and burn-ups.

At first inert matrices were tested without fissile material (EFTTRA T2 [5] and T2bis irradiation. [6]) From these early irradiation tests, $MgAl_2O_4$ (spinel) and $Y_3Al_5O_{12}$ emerged as suitable candidates as support material for actinide transmutation, due to their good neutron irradiation stability. The EFTTRA T3 experiment was designed to study the behaviour of UO_2 in various inert matrices, amongst which spinel. [7,8] Uranium was used as a simulant for americium, which is more difficult to handle. The EFTTRA T4 irradiation [9] focused on the behaviour of $^{241}AmO_x$ in spinel. As a follow-up experiment the EFTTRA T4bis irradiation [10] was started, which contained similar $^{241}AmO_x$/spinel targets, but which were irradiated to a higher burn-up (actinide depletion of 57%). The EFTTRA T4ter irradiation [10] (10 vol% UO_2 in spinel) especially assessed the thermal behaviour of spinel targets to a high burn-up, and was equipped with a central thermocouple.

Whereas the EFTTRA spinel irradiation focused on minor actinide transmutation, the OTTO (Once Through Then Out) experiment investigates the use of spinel as support material for plutonium incineration in a once through mode. The OTTO irradiation is a collaboration between PSI, JAERI and NRG. The irradiation test started in Autumn 2000 in the Petten HFR and is still ongoing. Four OTTO capsules (of a total of seven) consist of Pu-containing inclusions in spinel. The OTTO fuel is of a hybrid fuel type, i.e. the fissile material is embedded in ceramic inclusions, which themselves are contained in another inert matrix. Hybrid fuels, for OTTO $(Pu,Er/U,Y,Zr)O_{2-x}$ inclusions mixed with spinel, are at the moment considered to be one of the most realistic fuel types for real transmutation applications in LWRs.

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[1] EFTTRA, Experimental Feasibility of Targets for TRAnsmutation, is a collaboration of CEA, EdF, FZK, JRC-ITU, JRC-IE and NRG.

This paper describes the spinel irradiation tests, that have been performed at the HFR Petten for the transmutation of americium (EFTTRA collaboration) and plutonium in a once through mode (OTTO). From these tests a global picture emerges about the feasibility of using spinel inert matrix fuel. We discuss the various experiments and summarise the general picture of the characteristics of spinel inert matrix fuels.

Fabrication of spinel inert matrix pellets

An essential parameter in inert matrix fuels is the size of the fissile inclusion, because the irradiation damage to the matrix and its mechanical behaviour are directly related to the fissile particle size. [11] Depending on the size of the fissile inclusions we speak of micro-dispersed (diameter typically $d < 10$ micron) and macro-dispersed ($d \sim 50\text{-}250\ \mu m$) inert matrix fuels.

For the fabrication of heterogeneous CERCER fuel various fabrication routes can be distinguished, such as co-precipitation and low impact mixing of powders. With co-precipitation the starting materials are precipitated after a first dissolution step. Low impact powder mixing uses sinteractive powders of fissile and inert matrix material, which are mixed and pressed into pellets. Co-precipitation always yields sub-micron sized (i.e. micro-dispersed) fissile particles, whereas with powder mixing the size of the fissile inclusion depends on the grain size of the actinide powder and is typically $d \sim 10\text{-}250\ \mu m$ (macro-dispersed).

A third fabrication route, which yields sub-micron sized fissile particles, is based on the infiltration of aqueous metal solutions (containing the actinides) into porous inert matrix pellets. This so-called INRAM (Infiltration of Radio-Active Materials) process was developed at ITU, [12] and used to fabricate the Am-containing spinel targets for the EFTTRA T4 and T4bis experiments. [9]

The fabrication of hybrid fuels follows a more complicated route. The fissile inclusions are prepared separately using a sol-gel procedure. The spherical particles are then mixed with the inert matrix powder and the pellets are made in a similar way as with low impact powder mixing. With this method a far better control as to the final size of the (macro-dispersed) fissile particles is achieved. The second advantage of this fabrication route is the easy and clean handling of actinides. Therefore this fabrication route is considered the most promising for actual fuel fabrication. [11] Konings *et al.* [13] give an extensive description of various routes, used to fabricate inert matrix fuel.

Spinel irradiations at the HFR Petten

EFTTRA T3

From the EFTTRA T2 and T2bis irradiation [5,6] it was concluded that $MgAl_2O_4$ and $Y_3Al_5O_{12}$ are the most promising inert matrix materials. Al_2O_3 was discarded due to its large swelling and CeO_2 because of its incompatibility with the (stainless steel) cladding material. In the EFTTRA T3 a series of inert matrices were tested, without fissile material and with 2.5 vol% UO_2 and 19.6 vol% UY_6O_x inclusions. The uranium was 20% enriched in ^{235}U. Amongst the matrices under investigation were $MgAl_2O_4$ with UO_2 (micro- and macro-dispersed) and $MgAl_2O_4$ with macro-dispersed UY_6O_x. All targets had the same amount of fissile atoms per cm^3 matrix. The T3 targets were irradiated in the HFR for 198.9 full power days to a total fluence of $\sim 0.7 \cdot 10^{26}\ m^{-2}$ ($E > 0.1$ MeV). The irradiation parameters of the spinel targets in the T3 irradiation are listed in Table 1.

As there are both micro- and macro-dispersed targets in the T3 irradiation, the influence of the UO_2 particle size could be investigated. The volume of the irradiation damage induced by fission products is determined by a stopping range of typically 8-10 μm. Spinel with micro-dispersed UO_2 particles suffers from a more or less homogeneous irradiation damage in the whole matrix. In macro-dispersed (~250 μm) UO_2 the irradiation damage is confined largely to the fissile phase itself and in a small ring of ~10 μm thickness around the UO_2 particle. [14] In Figure 1 ceramographic images are shown of two EFTTRA T3 targets, with micro-dispersed UO_2 particles and with macro-dispersed particles.

The most remarkable difference is the heavy fracturing found in the macro-dispersed target, whereas the irradiated micro-dispersed target shows less porosity than the un-irradiated target. [8] Due to swelling of the UO_2 macro-particles, the surrounding spinel matrix fractures under high stresses from the swollen UO_2. The fractures run from fissile to fissile inclusion, which can be distinguished clearly in Figure 1b. An extensive description of the fracture behaviour in the macro-dispersed T3 targets is given in ref. [15]

Another distinct feature is the large fractional gas release (FGR) of macro-dispersed UO_2 in spinel, which is probably facilitated by the severe fracturing of the fuel pellets (cf. Table 1).

Table 1. **Irradiation parameters and results of the post irradiation examination of the spinel targets in the EFTTRA T3 experiment**

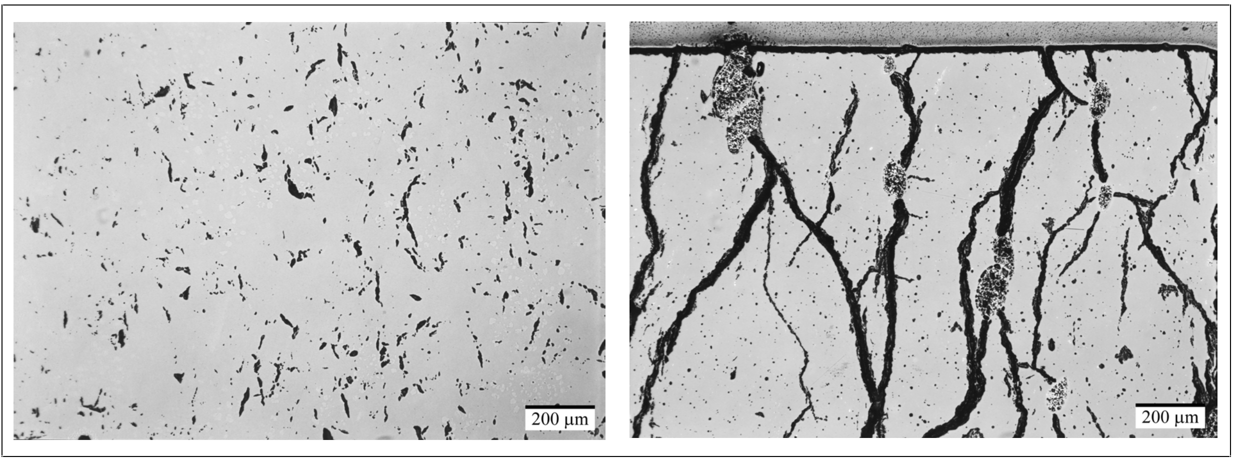
Irradiation	**EFTTRA T3, pin 9**		**EFTTRA T3, pin 11**		**EFTTRA T3, pin 13**	
Composition	7.3 wt% UO_2 in spinel (micro-dispersed)		7.6 wt% UO_2 in spinel (macro-dispersed)		31.2 wt% UY_6O_x in spinel	
Irradiation time	198.9 full power days		198.9 full power days		198.9 full power days	
Fluence (E >0.1 MeV)	$0.68 \cdot 10^{26}$ m^{-2}		$0.72 \cdot 10^{26}$ m^{-2}		$0.72 \cdot 10^{26}$ m^{-2}	
Burn-up	19.1 %FIMA		19.7 %FIMA		19.8 %FIMA	
Swelling	-0.8 ±1.3 vol%		6.5 ±5.6 vol%		2.9 ±0.8 vol%	
Fractional gas release	Kr	Xe	Kr	Xe	Kr	Xe
	0.3 ±0.1%	0.1 ±0.0%	48 ±5 %	43 ±5 %	3.2 ±0.3%	3.1 ±0.3%

The fracturing also explains the volume increase ~6.5% of the macro-dispersed UO_2 fuel. The micro-dispersed UO_2 show a far lower FGR and virtually no swelling, indicating that the gas retention properties of spinel are good. [14]

EFTTRA T4 and T4bis

The EFTTRA T4 and EFTTRA T4bis experiment were designed specifically to investigate the transmutation of americium. The irradiation targets, both from the same fabrication batch, consisted of 12.5 wt% $^{241}AmO_x$ in spinel (^{241}Am content of 11.2 wt%). They were irradiated at the HFR in Petten for 358.4 full power days (EFTTRA T4) and of 652.6 full power days (EFTTRA T4bis), to a total fast fluence (E > 0.1 MeV) of $\Phi = 1.68 \cdot 10^{26}$ m^{-2} and $\Phi = 2.13 \cdot 10^{26}$ m^{-2}, respectively. The most prominent characteristic of americium transmutation is the large production of helium (mostly from decay of ^{242}Cm, $t_{½} = 163$ days).

Figure 1. **Post irradiation ceramographic images of two EFTTRA T3 targets**



a) Pin 9, micro-dispersed UO_2 in spinel (light). The black spots are pores. Pin 9 showed no fracturing and a lower porosity compared to the un-irradiated target.

b) Pin 11, macro-dispersed UO_2 in spinel. The latter is characterised by heavy fracturing, induced by the swelling of the large UO_2 inclusions.

After irradiation, both the T4 and the T4bis capsule were examined non-destructively. The gas composition of the capsules and the swelling of the targets were investigated. In Table 2 the gas composition and fractional gas release of both T4 and T4bis irradiation are listed. Compared to the T4 irradiation the volumetric swelling of T4bis has increased to 29 vol%, which is due to the higher burn-up. Also the fractional gas release of both helium (48%) and the fission gases xenon and krypton (16%) into the plenum was very large. Possibly the porosity in the spinel targets has increased so much, that percolation paths were formed facilitating gas release into the plenum. The formation of open paths was not observed in the ceramographic images of T4, which showed only isolated bubbles. With the extra gas production due to the longer irradiation, the increased porosity may have facilitated the large release of both helium and fission gases. Destructive PIE on the T4bis pellets, which is foreseen in the near future,[2] can confirm if this is indeed the case.

EFTTRA T4ter

The T4ter-capsule contained 10vol% UO_2 (20% enriched in ^{235}U) in spinel. The targets were prepared by coprecipitation, which yielded sub-micron sized fissile inclusions in spinel. The T4ter targets were irradiated at the HFR in Petten for 652.6 full power days to a total fluence of $\Phi = 1.18 \cdot 10^{26}$ m^{-2} ($E > 0.1$ MeV). [10] The capsule was equipped with a central thermocouple. The UO_2 experienced a high burn-up of 32%FIMA, i.e. almost all ^{235}U and about 16% of the ^{238}U was incinerated. The fuel temperatures, as measured by the central thermocouple, were T ~1 000ºC at the beginning of irradiation, decreasing to 400ºC at the end of irradiation. The cladding temperature was kept approximately constant at T = 300ºC.

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[2] Currently the EFTTRA T4bis capsule is waiting to be transported to CEA Cadarache, where the destructive PIE will be performed.

Table 2. **Irradiation parameters and results of the post irradiation examination of the EFTTRA T4 and EFTTRA T4bis experiments**

Irradiation	**EFTTRA T4**			**EFTTRA T4bis**		
Composition	11.1 wt% ^{241}Am (as AmO_x) in spinel			11.2 wt% ^{241}Am (as AmO_x) in spinel		
Irrad. Time	358.4 full power days			652.6 full power days		
Fluence	$1.68 \cdot 10^{26}$ m^{-2} (E > 0.1 MeV)			$2.13 \cdot 10^{26}$ m^{-2} (E > 0.1 MeV)		
Depletion*	28 %			~57 %		
^{241}Am burn-up	96 %			99.8 %		
Swelling (max)	18 vol%			29 vol%		
Gas contents	Produced amount (mol)	Released amount (mol)	FGR (%)	Produced amount (mol)	Released amount (mol)	FGR (%)
He	$1.37 \cdot 10^{-3}$	$2.66 \cdot 10^{-4}$	19.5	$1.84 \cdot 10^{-3}$	$8.88 \cdot 10^{-4}$	48
Xe + Kr	$2.29 \cdot 10^{-4}$	$1.19 \cdot 10^{-5}$	5.2	$4.21 \cdot 10^{-4}$	$6.85 \cdot 10^{-5}$	16
Total	$1.60 \cdot 10^{-3}$	$2.78 \cdot 10^{-4}$	17.4	$2.27 \cdot 10^{-3}$	$9.56 \cdot 10^{-4}$	42

* The depletion is defined as (#actinides(BOI) - #actinides(EOI)) / #actinides(BOI) x 100%

The most important characteristic of the T4ter irradiation, was its large swelling of ~11 vol%, which was directed almost exclusively in radial direction. On the other hand, the destructive PIE revealed an unexpectedly small porosity. In Figure 2 a representative ceramographic picture of a T4ter target is shown. The porosity as observed from ceramography is about 0.2%. In addition a highly regular circular crack is observed. Especially the combination of a large swelling and a low porosity is remarkable. We discuss two possible explanations.

- The swelling is mainly caused by amorphisation of the spinel phase.

  Due to the small fissile particles the whole inert matrix is subject to highly energetic fission products, in contrast with the macro-dispersed targets, where only a small region of the inert matrix around the fissile phase is penetrated by fission products. Strong swelling of spinel was also observed in the THERMET irradiation, [16] in a spinel target with similarly micro-dispersed UO_2 inclusions as T4ter. However, the micro-dispersed T3 target with a lower UO_2 content did not show any significant swelling (cf. Table 1). Amorphisation of spinel and subsequent swelling due to highly energetic ion bombardment was also found by Wiss *et al.* [17] Swelling of spinel due to amorphisation, induced by fission products, is therefore a plausible explanation of the T4ter swelling behaviour.

- The T4ter target contains very small pores (< 5 µm), too small to be resolved by ceramography.

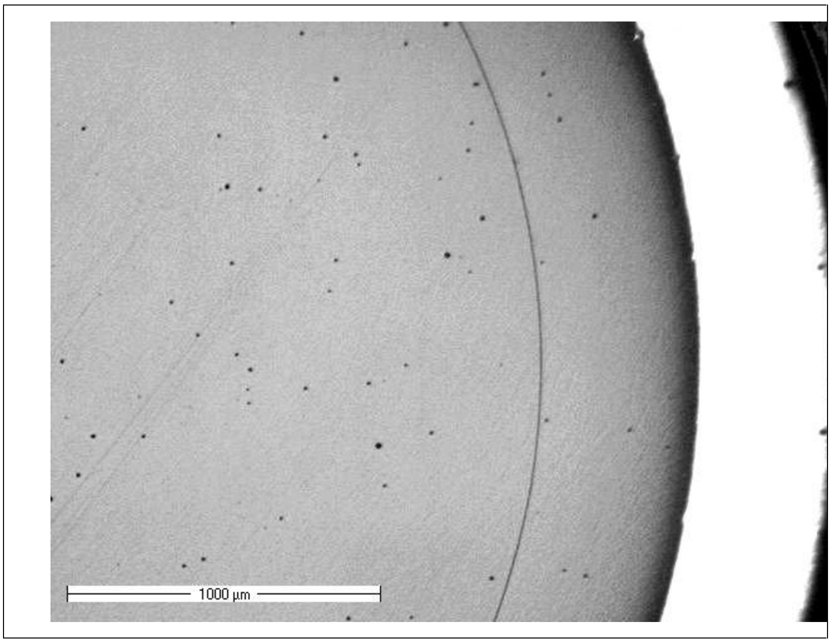
  In this scenario the produced fission gases are responsible for the swelling. The central temperature during irradiation (~1 000ºC at the beginning of irradiation and decreasing to ~400ºC) is too low to enable a large mobility of fission atoms. For example, extensive helium release in spinel was triggered only at temperatures larger than 1 100ºC. [18] Therefore it is not unlikely that fission gas atoms form small inclusions, smaller than the resolution of the optical microscope (~5 µm).

Unfortunately, the results of the gas puncturing and analysis, which was done after irradiation, did not give a reliable estimate about the fractional gas release in the T4ter irradiation, but the micro-dispersed T3 target had a very low FGR, however in combination with a negligible swelling.

Table 3. **Irradiation parameters of the EFTTRA T4ter experiment**

Irradiation	**EFTTRA T4ter**
Composition	25 wt% UO_2 in spinel (micro-dispersed)
Irradiation time	652.6 full power days
Fluence (E > 0.1 MeV)	$1.18 \cdot 10^{26}$ m^{-2}
Burn-up	32 %FIMA
Swelling	10.9 ±1.5 vol%
Fractional gas release	No results available

Figure 2. **Ceramographic image of an irradiated T4ter pellet** (detail)



The white rim at the right is the steel cladding. The large swelling of the pellet has completely closed the gas gap. Furthermore the picture shows a remarkable circular crack in the matrix and unexpectedly low porosity.

OTTO

The OTTO targets containing spinel were made by PSI and NRG and consisted of 20 vol% $(Pu,U,Y,Zr)O_{2-x}$ and $(Pu,Er,Y,Zr)O_{2-x}$ inclusions in spinel.[3] The Pu-containing particles were prepared by the internal gelation procedure. The size of the inclusions varies between d < 25 µm and d ~200-250 µm. This kind of hybrid fuel concept is closer to real fuel application in LWR's than the EFTTRA fuels, which were purely designed for the testing of the spinel matrix. The OTTO irradiation started on 26 October 2000 and is expected to end on 31 December 2002.

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[3] The OTTO irradiation consisted of seven targets, four of which contained spinel as inert matrix. The three other capsules were two solid solution targets, based on zirconia, and a reference MOX capsule. These targets were fabricated by PSI.

During a sintering test at 1 700ºC, unexpectedly a severe degradation was found of the Pu-phase spheres that were mixed with spinel. [19] As the melting point of spinel is 2 135ºC, the mixture of spinel and $(Pu,U/Er,Y,Zr)O_{2-x}$ has apparently a much lower melting point. This behaviour may be of importance and raises questions about the applicability of spinel as an inert matrix for Pu-incineration. On the other hand, from neutrographic imaging during irradiation, the volumetric swelling of the OTTO targets was found to be limited to a few percent, even after 14 cycles (~350 full power days) of irradiation (burn-up ~120 GWd/m^3). Also the fuel temperatures, as measured by central thermocouples, which started at ~1 000ºC and decreased to currently ~800ºC, suggest good irradiation behaviour. The cladding temperature is about 400ºC.

Although the OTTO irradiation seems to progress well, the unexpected melting-like behaviour of spinel-based targets at reasonably low temperatures (~1 700ºC) is worrying. Note that Nitani *et al.* [20] observed a decomposition of spinel during irradiation in the case of U-ROX fuel, at temperatures higher than ~1 427ºC (1 700 K). These temperatures put a severe limit on the operational temperatures of spinel-based targets.

These observations make clear that a careful study should be made about the compatibility of spinel with Pu-containing fissile inclusions and the stability of (irradiated) $MgAl_2O_4$ at high temperatures. Also leaching aspects of spinel with fissile inclusions have to be assessed, when spinel is to be used as a matrix in a once-through mode.

Discussion

After extensive irradiation testing of $MgAl_2O_4$ spinel, a general picture arises about the properties and characteristics of spinel under irradiation conditions. In the discussion we address two issues.

- Micro-dispersed versus macro-dispersed fissile inclusions.

  From the T4ter irradiation it was found that sub-micron sized fissile inclusions cause extensive swelling, [10] in line with observations of the THERMET irradiation [16] and heavy-ion irradiation studies [17]. Macro-sized inclusions cause heavy fracturing of the matrix. Whether the swelling of spinel is caused by fission gas pressure, or by fission product damage and associated amorphisation of the matrix material, is still an unresolved question.

  A volumetric swelling of the co-precipitated EFTTRA T3 target (pin 9) was not observed, but this target had a reasonable porosity before irradiation (density 90% T.D.) and a small volume fraction of UO_2 inclusions. It seems that the initial porosity has accommodated the solid state swelling of the spinel, so that no volume increase was observed. [15]

  For micro-dispersed fuel targets an increased initial porosity could be able to keep the volumetric swelling within acceptable limits. The same holds for macro-dispersed CERCER fuel. However, in these targets the fissile macro-particle should have an initial porosity, large enough to accommodate the swelling within the inclusion. This may avoid the heavy fracturing of the inert matrix material observed in macro-dispersed fuel targets.

- Spinel as matrix for americium transmutation or for plutonium burning.

  For the transmutation of americium the accommodation of large amounts of helium is the most important issue. Typically in americium transmutation the amount of gas produced per initial fission atom is ten times larger than in UO_2. In the EFTTRA T4 / T4bis irradiations a large swelling and a large porosity is observed in combination with a large fractional gas release. However, these were micro-dispersed AmO_x inclusions, which cause irradiation damage in the whole spinel matrix. Retention of helium in the matrix is preferable to reduce the plenum volumes required in fuel pins, but then the swelling of the matrix should also be limited or controlled. Future irradiation tests with americium fuel are required to reach a definite fuel concept for americium transmutation.

The OTTO irradiation studies the feasibility of spinel (and of zirconia-based targets) for plutonium burning. Due to the macro-dispersed fissile inclusions, the swelling is limited. The thermal behaviour is moderate and in line with FEM calculations, with temperatures typically lower than 1 000-1 100ºC. The post irradiation examination of OTTO, which is foreseen to start in the spring of 2003, should give further information about how the inert matrix has behaved during irradiation. Apart from the irradiation behaviour, the chemical stability of spinel in combination with plutonium at high temperatures remains an important issue.

Conclusions

At the HFR Petten extensive irradiation testing of spinel inert matrix fuels has been performed, for the transmutation of americium (EFTTRA programme) and also for Pu-ROX fuel (OTTO). In recent years, different irradiations were performed on spinel, with UO_2 fissile phases used as simulant, AmO_x (EFTTRA T4 and T4bis) and hybrid plutonium fuel $(Pu,U/Er,Y,Zr)O_{2-x}$ (OTTO). A general picture has emerged about the properties of spinel under various irradiation conditions. The swelling under the influence of fission and the instability of spinel at high temperatures are serious drawbacks, which may ultimately result in the rejection of spinel as potential inert matrix material. Due to this problems we observe a shift towards different inert matrix materials, and towards specific fuel concepts, designed for the use in LWR's. For example, the EFTTRA T5 irradiation, which is being designed at the moment , [21] is aiming at using hybrid fuel concepts and the embedding of actinides in solid solutions (specifically zirconia) for the transmutation of americium.

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