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# Fission product release from ZrC-coated fuel particles during post-irradiation heating at 1800 and 2000°C

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## Abstract

The ZrC coating layer is a candidate to replace the SiC coating layer of the Triso-coated fuel particles for high-temperature gas-cooled reactors. Post-irradiation heating tests of the ZrC–Triso coated $UO_2$ particles were performed at 1800°C for 3000 h and at 2000°C for 100 h to study the release behavior of fission products. The fission gas release monitoring and the X-ray microradiography revealed that no through-coating failure occurred during the heating tests. The high cesium retention of the ZrC–Triso coated fuel particles was confirmed up to 1800°C. The diffusion coefficient for cesium in the ZrC layer was more than two orders smaller than that in the SiC layer at 1800°C. The diffusion coefficient for ruthenium in the ZrC layer was almost the same as that for cesium in the SiC layer. © 1997 Elsevier Science B.V.

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## 1. Introduction

The high temperature gas-cooled reactor (HTGR) uses coated fuel particles, which are dispersed in a graphite matrix to form a fuel element. In current HTGR designs the Triso-coated fuel particles are to be used [1–4]. The Triso-coated fuel particle consists of a microspherical kernel of oxide or oxycarbide fuel and coating layers of porous pyrolytic carbon (PyC), inner dense PyC (IPyC), silicon carbide (SiC) and outer dense PyC (OPyC). The function of these coating layers is to retain fission products within the particle. The porous PyC coating layer, called the buffer layer, attenuates fission recoils and provides void volume for gaseous fission products and carbon monoxide. The IPyC coating layer acts as a containment to gases. The SiC coating layer provides mechanical strength for the particle and acts as a barrier to the diffusion of metallic fission products which diffuse easily through the IPyC layer. The OPyC coating layer protects the SiC coating layer mechanically.

Although SiC has excellent properties, it gradually loses mechanical integrity at very high temperatures, especially above 1700°C, by thermal dissociation [5–7], leading to extensive release of fission products from the Triso-coated fuel particles. The fuel temperatures are, therefore, limited to well below 1700°C even in the design-basis accidents in the current HTGR designs [2–4].

Zirconium carbide (ZrC) is known as a refractory and chemically stable compound, having a melting point of 3540°C and melts eutectically with carbon at 2850°C [8]. The ZrC coating layer is a candidate to replace the SiC coating layer of the Triso-coated fuel particles; the resulting particle is termed a ZrC–Triso coated fuel particle. The review and evaluation of the ZrC coating layer for HTGR fuel particles have been made by Kasten et al. [9] and Minato et al. [10]. The results of irradiation experiments of the ZrC-coated fuel particles were encouraging [8,11–13]. It has been demonstrated that the ZrC–Triso coated fuel particles have a much higher temperature stability than the normal Triso-coated fuel particles [8,13]. In addition, the ZrC coating layers have higher resistance to chemical attack by fission product palladium than the SiC coating

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layers [14,15]. For the retention of fission products by the ZrC coating layer, however, the data are limited [16–18].

The present work was part of a series of the post-irradiation heating experiments of the ZrC–Triso coated fuel particles. In the previous post-irradiation heating test at 1600°C for 4500 h, the ZrC coating layer showed better cesium retention than the SiC coating layer though the ZrC layer was a less effective barrier to ruthenium [16]. In the present work the post-irradiation heating tests of the ZrC–Triso coated fuel particles at 1800°C for 3000 h and at 2000°C for 100 h were performed to study the release behavior of fission products.

## 2. Experimental

### 2.1. Samples

The ZrC–Triso coated fuel particles used in the present experiments were sampled from the same irradiated fuel compact that was used in the previous heating experiment [16]. The fuel compact was deconsolidated electrolytically after the irradiation. Table 1 shows the characteristics of the ZrC–Triso coated fuel particles. The particles had $UO_2$ kernels and the ZrC coating layer was deposited by the bromide process [19]. Fig. 1 shows optical micrographs of a typical polished cross-section of the irradiated ZrC–Triso coated fuel particle before heating experiment.

The ZrC–Triso coated fuel particles were irradiated in the form of an annular fuel compact loaded in a gas-swept capsule in the Japan materials testing reactor (JMTR). The irradiation conditions are shown in Table 2 [13]. The burnup of the fuel was 1.5% FIMA and the irradiation temperature was 900°C. The release of short-lived noble gases was measured during irradiation. The release-to-birth ratio of $^{88}$Kr ($T_{1/2} = 2.84$ h) during irradiation was from $2 \times 10^{-7}$ to $4 \times 10^{-7}$ at about 900°C [13], which indicated that no failed particle was contained in the fuel compact. The release could be ascribed to the uranium contamination of the graphite matrix [20].

Table 1
Characteristics of ZrC–Triso coated fuel particles

| | Diameter or thickness (μm) | Density (Mg/m$^3$) |
|---|---|---|
| $UO_2$ kernel | 608 | 10.6 |
| Buffer layer | 64 | 1.11 |
| IPyC layer | 26 | 1.84 |
| ZrC layer | 31 | 6.6 |
| OPyC layer | 55 | 1.95 |

### 2.2. Post-irradiation heating

The equipment used for the present post-irradiation heating tests was the same as used previously [16]. The furnace, which is installed in a hot cell, is composed of a graphite heater, a graphite sample holder, graphite holder disks and carbon insulators within a stainless steel vessel. For each heating test, 100 of the ZrC–Triso coated fuel particles were placed individually in the holes of two graphite disks and heated in the flowing helium. Before heating, the coated fuel particles were examined by X-ray microradiography to confirm the mechanical integrity of the coating layers. The temperature was read and controlled with an optical pyrometer from the top of the furnace.

Two heating tests at 1800°C for 3000 h and at 2000°C for 100 h were performed. As shown in Table 3, the tests were divided into eight and four time steps, respectively. At the end of each step the graphite components and the carbon insulators were replaced by new ones for measurements of the released metallic fission products. The coated fuel particles were X-ray microradiographed to examine the integrity of the coating layers and then heated repeat-

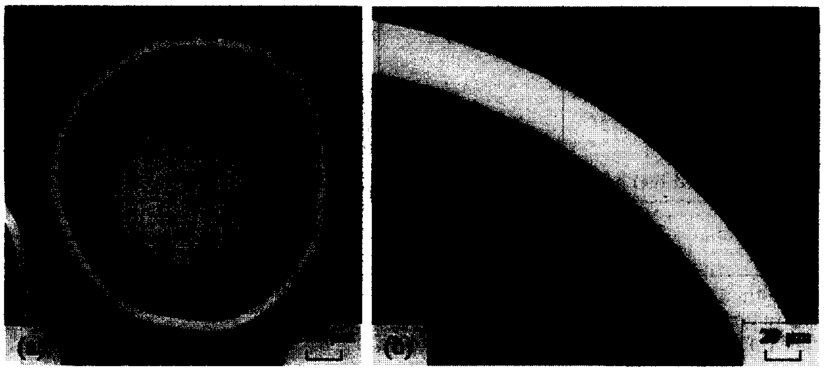

Fig. 1. Optical micrographs of polished cross-section of irradiated ZrC–Triso coated fuel particle before heating test.

Table 2
Irradiation conditions of ZrC–Triso coated fuel particles

| | |
|---|---|
| Duration | 79.9 EFPD |
| Temperature | 900°C |
| Burnup | 1.5%FIMA |
| Fluence | $1.2\times10^{25}$ $m^{-2}$ (E > 29 fJ) |
| $R/B(^{88}FUNCKr)$ | $2\times10^{-7}$ to $4\times10^{-7}$ |

edly for 3000 or 100 h. During heating, the fission gas release of $^{85}$Kr ($T_{1/2}$ = 10.73 y) in the flowing helium was monitored by an ionization chamber.

### 2.3. Post-heating examinations

The graphite components and the carbon insulators, which were removed from the furnace at the end of each time step, were measured by gamma-ray spectrometry to identify and evaluate the released fission products. The fission product release data were treated as the fractional release:

$$F = R/I, \qquad (1)$$

where $F$ is the fractional release of a nuclide, $R$ the released amount of the nuclide measured by gamma-ray spectrometry and $I$ the inventory of the nuclide measured before the heating tests.

All the ZrC–Triso coated fuel particles heated in each test were examined by X-ray microradiography. Fifty particles sampled from each test were embedded in epoxy resin and polished to the equator of the particle for optical microscopy.

## 3. Results and discussion

### 3.1. Coating integrity

After heating at 1800°C for 3000 h, no anomaly was found in the coating layers and fuel kernels by X-ray microradiography of the ZrC–Triso coated fuel particles, except some signs of the IPyC coating failure in ten out of 100 particles. Typical polished cross-sections of the ZrC–Triso coated fuel particles after heating at 1800°C for 3000 h are shown in Fig. 2. No failure was observed in the ZrC and OPyC coating layers on the polished cross-sections. However, some degradation of the ZrC coating layer seemed to have occurred. The inner and outer surfaces of the ZrC coating layers in most of the particles heated at 1800°C were not smooth, which was in contrast with the case of the particles before heating as shown in Fig. 1. In some particles the IPyC coating layers were cracked in the radial direction, where about a quarter of the thickness of the ZrC coating layers seemed to be attacked along the grain boundaries.

Fig. 3 shows typical examples of X-ray microradiographs of the ZrC–Triso coated fuel particles after heating at 2000°C for 100 h. Only seven out of 100 particles showed no anomaly in the X-ray microradiographs, as shown in Fig. 3(a). The rest of the particles had various anomalies in the coating layers and fuel kernels. The failure of the IPyC coating layers and the migration of the fuel material are seen in Fig. 3(b)–(d). However, no failure of the OPyC coating layer was found.

The ceramography of the ZrC–Triso coated fuel particles after heating at 2000°C for 100 h revealed that the ZrC coating layers were damaged as well as the IPyC coating layers in most of the particles observed, as shown in Fig. 4. The ZrC coating layers were damaged through the thickness. The appearance of the particles in Fig. 4 was very similar to that of the ZrC–Triso coated fuel particles heated to 2400°C at the ramp rate of 1°C/min [13].

The X-ray microradiography and ceramography of the ZrC–Triso coated fuel particles suggest that the degradation of the ZrC coating layer be related to the failure of the IPyC coating layer. An electron probe microanalysis of the polished cross-section of the particles and a thermochemical analysis in a relevant system, which are under consideration, will probably reveal mechanisms of the degradation of the coating layers.

### 3.2. Release of fission gas

During both the heating tests at 1800°C for 3000 h and at 2000°C for 100 h, no burst release of fission gas was

Table 3
Heating conditions of ZrC-Triso coated fuel particles

| | | Step number | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| At 1800°C | step time (h) | 24 | 46 | 80 | 200 | 550 | 700 | 900 | 500 |
| | cumulative time (h) | 24 | 70 | 130 | 350 | 900 | 1600 | 2500 | 3000 |
| At 2000°C | step time (h) | 10 | 14 | 14 | 62 | | | | |
| | cumulative time (h) | 10 | 24 | 38 | 100 | | | | |

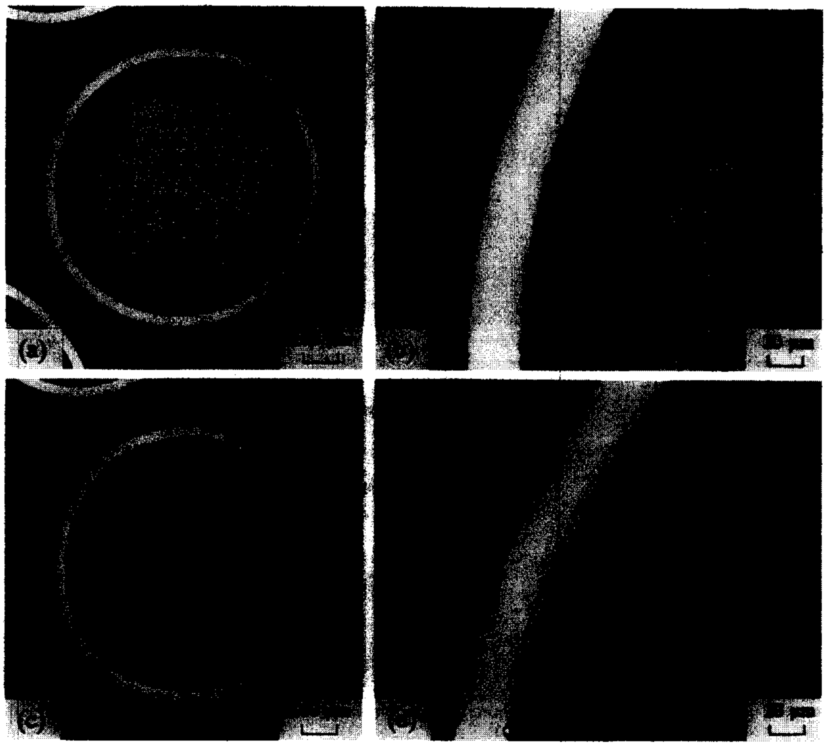

Fig. 2. Optical micrographs of polished cross-section of irradiated ZrC–Triso coated fuel particle after heating at 1800°C for 3000 h.

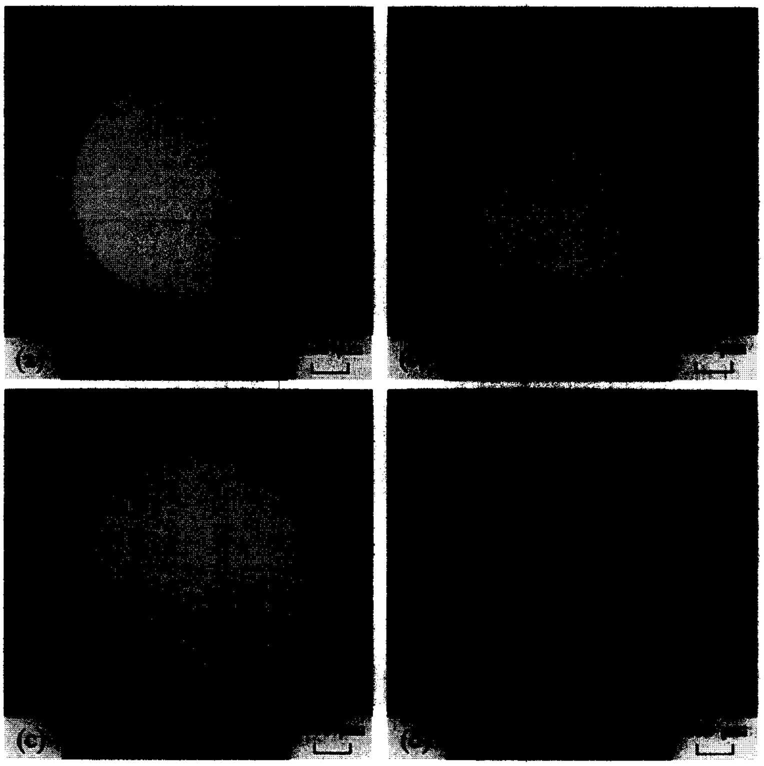

Fig. 3. X-ray microradiographs of irradiated ZrC–Triso coated fuel particles after heating at 2000°C for 100 h.

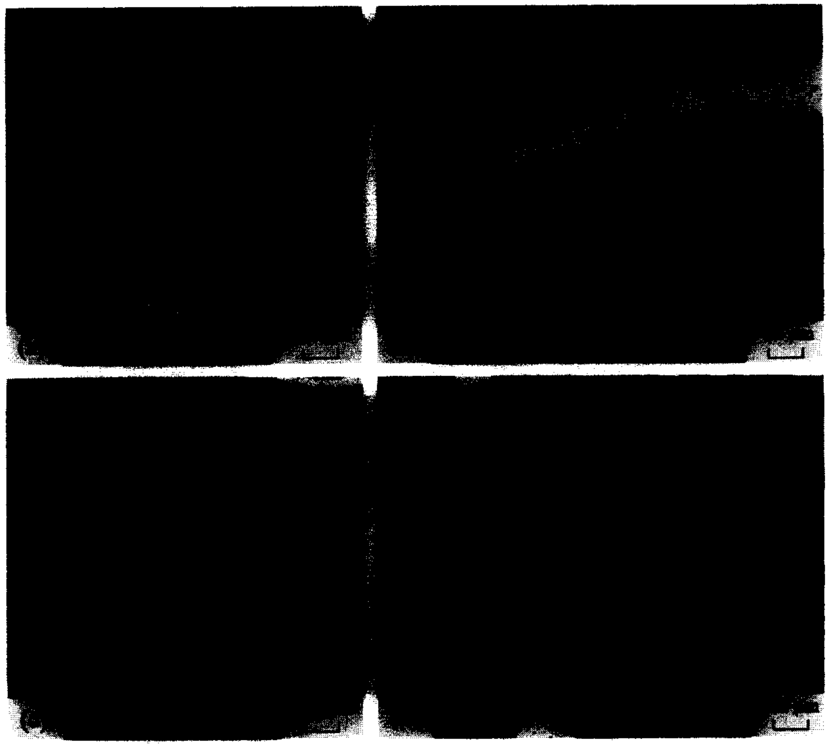

Fig. 4. Optical micrographs of polished cross-section of irradiated ZrC–Triso coated fuel particles after heating at 2000°C for 100 h.

detected by the $^{85}$Kr release monitoring. The fission gas was kept in the ZrC–Triso coated fuel particles during both the heating tests.

In the ZrC–Triso coated fuel particles, fission gas would be released only when all the coating layers of IPyC, ZrC and OPyC fail. This kind of coating failure is called the through-coating failure. No fission gas release in the present heating tests means that no through-coating failure occurred in the ZrC–Triso coated fuel particles. The X-ray microradiographs of the coated fuel particles after the heating tests revealed no OPyC failure, which was confirmed by the results of the gas release monitoring.

### 3.3. Release of metallic fission products

#### 3.3.1. Release of cesium

The gamma-ray spectrometry of the graphite components and carbon insulators revealed that $^{134}$Cs and $^{137}$Cs were released from the particles. The measured fractional releases of $^{137}$Cs at 1800°C for 3000 h and at 2000°C for 100 h are shown in Fig. 5 as a function of heating time, where the broken curves are smooth fits to the data points. The data of measured fractional release of $^{137}$Cs at 1600°C for 4500 h in the previous test [16] are also presented in Fig. 5 for comparison. It was found that the fractional release of $^{137}$Cs was below $1 \times 10^{-3}$ even after heating at 1800°C for 3000 h, whereas it was more than $1 \times 10^{-1}$ after heating at 2000°C for 100 h. The release behavior of $^{134}$Cs was almost the same as that of $^{137}$Cs.

The effective diffusion coefficient for $^{137}$Cs in the ZrC coating layer, $D_{Cs}$(ZrC), was evaluated based on a diffu-

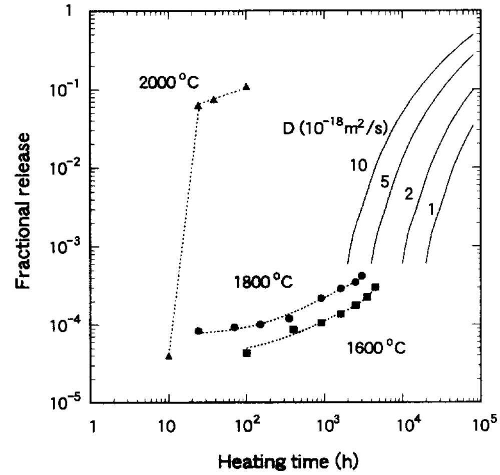

Fig. 5. Fractional release of $^{137}$Cs during post-irradiation heating of ZrC–Triso coated fuel particles as a function of heating time. Solid curves are calculated by a model of diffusion in one-layer coating. The data at 1600°C were quoted from Ref. [16].

sion model, where a fuel kernel with a single coating layer was assumed. In the calculation, a single coating layer was considered to be a diffusion barrier but a fuel kernel was not. This assumption is valid since the ZrC coating layer is a much better diffusion barrier to cesium than the PyC coating layers and $UO_2$ kernel. Further details of the analytical model were described elsewhere [16]. Based on the model, four diffusion release curves are also drawn in Fig. 5.

It is apparent in Fig. 5 that the shape of the measured fractional release curve at 1800°C was different from that of the calculated ones. The rather flat part of the measured curve at very low fractional release was attributed to the release of contaminated $^{137}$Cs in the OPyC layer, which was not considered in the calculation. The measured curve probably shows that the diffusion release through the ZrC layer just started near the end of the heating test. The fractional release curves of this kind were often observed in several post-irradiation heating experiments [16,21–23].

It could be, therefore, obtained from Fig. 5 that $D_{Cs}$(ZrC) at 1800°C lies between $2 \times 10^{-18}$ and $1 \times 10^{-17}$ m$^2$/s. In the previous experiment $D_{Cs}$(ZrC) at 1600°C was evaluated to be between $1 \times 10^{-18}$ and $5 \times 10^{-18}$ m$^2$/s [16]. The sudden increase in the fractional release at 2000°C was probably attributed to the degradation of the ZrC coating layer observed in the ceramography.

### *3.3.2. Release of ruthenium*

The measured fractional releases of $^{106}$Ru at 1800°C for 3000 h and at 2000°C for 100 h are shown as a function of heating time in Fig. 6, where the data of measured fractional release of $^{106}$Ru at 1600°C for 4500 h in the previous test [16] are also presented for comparison. The fractional release of $^{106}$Ru was about $9 \times 10^{-1}$ both at 1800°C for 3000 h and at 2000°C for 100 h.

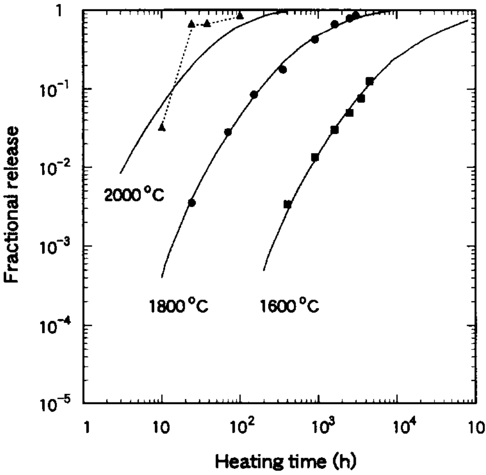

Fig. 6. Fractional release of $^{106}$Ru during post-irradiation heating of ZrC–Triso coated fuel particles as a function of heating time. Solid curves are calculated by a model of diffusion in a fuel kernel and one-layer coating. The data at 1600°C were quoted from Ref. [16].

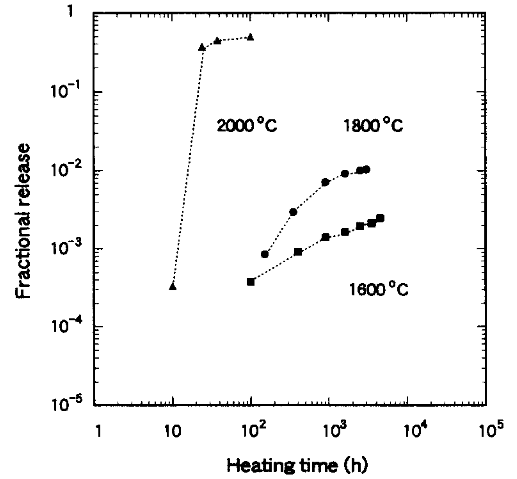

Fig. 7. Fractional release of $^{154}$Eu during post-irradiation heating of ZrC–Triso coated fuel particles as a function of heating time. The data at 1600°C were quoted from Ref. [16].

The release behavior of $^{106}$Ru was evaluated by the same diffusion model that used for the evaluation of $^{137}$Cs release, where a fuel kernel with a single coating layer was assumed. In contrast with the $^{137}$Cs release controlled by the transport only in the ZrC coating layer, the $^{106}$Ru release was determined by the transport both in the $UO_2$ kernel and in the ZrC coating layer. Both the diffusion coefficients for $^{106}$Ru in the $UO_2$ kernel, $D_{Ru}(UO_2)$, and in the ZrC coating layer, $D_{Ru}$(ZrC), were parameters in the model [16]. For the best fit as shown in Fig. 6, the diffusion coefficients of $D_{Ru}(UO_2) = 1 \times 10^{-15}$ m$^2$/s and $D_{Ru}(ZrC) = 5 \times 10^{-15}$ m$^2$/s at 1800°C were obtained.

The measured fractional release curve at 2000°C did not fit the calculated diffusion release curve, as shown in Fig. 6, which was drawn based on the extrapolated diffusion coefficients, $D_{Ru}(UO_2) = 2 \times 10^{-14}$ m$^2$/s and $D_{Ru}(ZrC) = 5 \times 10^{-14}$ m$^2$/s, from the data at 1600 and 1800°C. The sudden increase in the measured fractional release and the large fractional release compared with the calculated curve suggest the deterioration of the ZrC coating layer.

### *3.3.3. Release of other fission products*

Besides $^{137}$Cs, $^{134}$Cs and $^{106}$Ru, the release of fission products of $^{144}$Ce, $^{154}$Eu and $^{155}$Eu were detected by gamma-ray spectrometry of the graphite components and carbon insulators. The measured fractional releases of $^{154}$Eu at 1800°C for 3000 h and at 2000°C for 100 h are shown in Fig. 7 as a function of heating time, together with those at 1600°C for 4500 h. The fractional releases of $^{154}$Eu were larger than those of $^{137}$Cs at any temperature. Although the release data of $^{137}$Cs and $^{106}$Ru could be treated quantitatively, the accuracy of the measured values for $^{134}$Cs, $^{144}$Ce, $^{154}$Eu and $^{155}$Eu was not enough to evaluate the effective diffusion coefficients since the counts by gamma-ray spectrometry were small.

The radionuclide $^{110m}$Ag, whose half life is 250.4 d, is known as one of the most releasable radionuclides from the normal Triso-coated fuel particles. The release of $^{110m}$Ag was not detected in the present experiment. However, as mentioned in the previous paper [16], it cannot be concluded that the ZrC coating layer has excellent $^{110m}$Ag retentivity. The present experiment was carried out about 9 years after the end of irradiation of the ZrC-Triso coated fuel particles and the ratio of inventories of $^{110m}$Ag to $^{106}$Ru was less than $1 \times 10^{-3}$ just before the heating tests, suggesting that the inventory of $^{110m}$Ag was too small to be detected even if $^{110m}$Ag was released.

### *3.4. Comparison with normal Triso-coated fuel particles*

For the retention of fission gas, no through-coating failure of the ZrC–Triso coated fuel particles causing the fission gas release was observed in the present post-irradiation heating tests at 1800°C for 3000 h and at 2000°C for 100 h. In the case of the normal Triso-coated fuel particles, fission gas release was observed in the post-irradiation heating of fuel elements at 1800°C for 100 or 200 h [22,23]. Although the number of the particles tested in the present experiment was small compared with that in the fuel element, it is probably safe to say that the ZrC–Triso coated fuel particles have higher capability of fission gas retention than the normal Triso-coated fuel particles at high temperatures. This is supported by the results of the ramp tests of the ZrC–Triso and normal Triso-coated fuel particles [13,24]; one ZrC–Triso coated fuel particle out of 101 particles failed to release fission gas at 2400°C, whereas most of the normal Triso-coated fuel particles failed at that temperature.

For the retention of metallic fission products, fractional release of one of the key fission products of cesium from the ZrC–Triso coated fuel particles was less than $1 \times 10^{-3}$ at 1800°C for 3000 h, though more than $1 \times 10^{-1}$ was released at 2000°C for 100 h. The reported values for fractional release of cesium from the normal Triso-coated fuel particles during post-irradiation heating at 1800°C were more than $1 \times 10^{-1}$ for less than 100 h [21–23]. The high cesium retentivity of the ZrC–Triso coated fuel particles was confirmed up to 1800°C. Fig. 8 compares the diffusion coefficients for $^{137}$Cs in the ZrC layer with those in the SiC layer [21,25,26]. At 1800°C $D_{\mathrm{Cs}}(\mathrm{ZrC})$ was more than two orders smaller than $D_{\mathrm{Cs}}(\mathrm{SiC})$.

As estimated from the previous experimental results [16,17], the fractional release of $^{106}$Ru was larger than that of $^{137}$Cs from the ZrC–Triso coated fuel particles in the present experiment. The release of $^{106}$Ru was not reported in the post-irradiation heating tests of the normal Triso-coated fuel particles at 1600°C as long as 500 h and at 1800°C as long as 200 h [21–23]. The retention of $^{106}$Ru in the ZrC–Triso coated fuel particles was not better than that in the normal Triso-coated fuel particles. The diffusion coefficient of $D_{\mathrm{Ru}}(\mathrm{ZrC})$ obtained was almost the same as that of $D_{\mathrm{Cs}}(\mathrm{SiC})$ in the literature, as shown in Fig. 8.

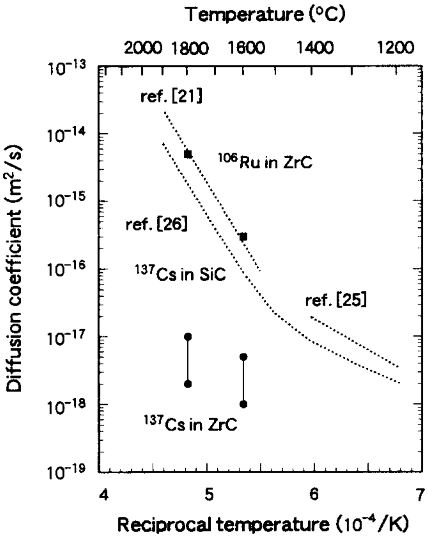

Fig. 8. Diffusion coefficients of $^{137}$Cs and $^{106}$Ru in ZrC obtained in the present and previous [16] experiments shown by solid symbols, together with those of cesium in SiC quoted from Refs. [21,25,26] shown by broken lines.

## 4. Conclusions

Post-irradiation heating tests of the ZrC–Triso coated $UO_2$ particles were performed at 1800°C for 3000 h and at 2000°C for 100 h to study the release behavior of fission products. The following was concluded.

(1) The fission gas release monitoring and the X-ray microradiography revealed that no through-coating failure occurred during the heating tests.

(2) The high cesium retention of the ZrC–Triso coated fuel particles was confirmed up to 1800°C. The diffusion coefficient for cesium in the ZrC layer was more than two orders smaller than that in the SiC layer at 1800°C.

(3) The diffusion coefficient for ruthenium in the ZrC layer was almost the same as that for cesium in the SiC layer.

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