POST IRRADIATION EXAMINATION OF THORIA-PLUTONIA MOX FROM AC-6 CLUSTER

by
Post Irradiation Examination Division
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Bhabha Atomic Research Centre
Mumbai, India
2014
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<th>No.</th>
<th>Description</th>
<th>Details</th>
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<tr>
<td>01</td>
<td>Security classification</td>
<td>Unclassified</td>
</tr>
<tr>
<td>02</td>
<td>Distribution</td>
<td>External</td>
</tr>
<tr>
<td>03</td>
<td>Report status</td>
<td>New</td>
</tr>
<tr>
<td>04</td>
<td>Series</td>
<td>BARC External</td>
</tr>
<tr>
<td>05</td>
<td>Report type</td>
<td>Technical Report</td>
</tr>
<tr>
<td>06</td>
<td>Report No.</td>
<td>BARC/2014/E/003</td>
</tr>
<tr>
<td>07</td>
<td>Part No. or Volume No.</td>
<td></td>
</tr>
<tr>
<td>08</td>
<td>Contract No.</td>
<td></td>
</tr>
<tr>
<td>09</td>
<td>Title and subtitle</td>
<td>Post irradiation examination of Thoria-Plutonia MOX fuel from AC-6 cluster</td>
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<tr>
<td>10</td>
<td>Collation</td>
<td>25p., 13 figs., 1 tab.</td>
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<tr>
<td>11</td>
<td>Project No.</td>
<td></td>
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<tr>
<td>12</td>
<td>Personal author(s)</td>
<td>Prema Mishra; J.L. Singh; J.S. Dubey; K.M. Pandit; V.P. Jathar; B.N. Rath; P.M. Satheesh; R.S. Shriwastaw; Priti Kotak Shah; Anil Bhandekar; Sunil Kumar; P.B. Kondejkar; H.N. Singh; S. Anantharaman</td>
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<td>13</td>
<td>Affiliation of author(s)</td>
<td>Post Irradiation Examination Division, Bhabha Atomic Research Centre, Mumbai</td>
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<td>14</td>
<td>Corporate author(s)</td>
<td>Bhabha Atomic Research Centre, Mumbai - 400 085</td>
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<tr>
<td>15</td>
<td>Originating unit</td>
<td>Post Irradiation Examination Division, Bhabha Atomic Research Centre, Trombay, Mumbai- 400 085</td>
</tr>
<tr>
<td>16</td>
<td>Sponsor(s) Name</td>
<td>Department of Atomic Energy</td>
</tr>
<tr>
<td>17</td>
<td>Type</td>
<td>Government</td>
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ThO₂-PuO₂, मिश्र ऑक्साइड ईधन समूह का पथ किरण परीक्षण

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पथ किरण परीक्षण प्रभाव
भाभा परमाणु अनुसंधान केंद्र

सार

भारत देश में सूर्यनीयम की अपेक्षा थोरियम के करीब चार गुना ज्यादा भंडार है। थोरियम का न्यूक्लियर ईधन के रूप में इस्तेमाल, भारतीय न्यूक्लियर उर्जा कार्यक्रम का प्रमुख अंग है। ThO₂-PuO₂ मिश्र ऑक्साइड ईधन अभ्यासी भारतीय दांति भारी पानी संग्रह (AHWR) में उपयोग के लिए विकसित किया जा रहा है। इस ईधन के विकास के लिए इसका निर्माण ऐयम शोध कार्य किया जा रहा है। एक ईधन समूह का जिससे (Th-4%Pu)O₂ ईधन था, CIRUS परमाणु संयंत्र में 18.5GW/Te उपयोग के उपरांत पथ किरण परीक्षण दिखाया गया। इस परीक्षण में ध्वंसात्मक और उसके बिना विभिन्न पद्धतियों का उपयोग किया गया। विभिन्न पद्धतियों में चाकुश प्रत्येकीकरण, पराध्यालित परीक्षण, आवर्त प्रवाह परीक्षण, गामा किरण क्रमवीक्षण, विमोचित विकंडन गैस विश्लेषण, सुरक्षा दर्शकीय ऐयम SEM शामिल हैं।

इस सभी मूल्यांकन और परीक्षणों को इस विवरण में प्रस्तुत किया गया है | परीक्षण परिणाम दर्शाते हैं कि ThO₂-PuO₂ मिश्र ऑक्साइड ईधन का कार्य-निष्पादन UO₂ ईधन से बेहतर है।
Post Irradiation Examination of Thoria-Plutonia MOX fuel from AC-6 cluster


Post Irradiation Examination Division,
Bhabha Atomic Research Centre,
Trombay, Mumbai-400085

Abstract

India has about four times more thorium resources than uranium. Utilisation of thorium for large scale energy production is a major goal in the three stage nuclear power programme. Thoria based mixed oxide is the candidate fuel for the Advanced Heavy Water Reactor (AHWR) being developed for thorium utilisation. Hence, research and development in fabrication, characterisation and irradiation testing of thoria based fuels is necessary. A fuel pin cluster consisting of (Th-4% Pu)O2 MOX fuel pins, was irradiated in the pressurised water loop (PWL) of CIRUS reactor to a peak fuel burnup of 18.5 GWd/Te. The peak linear heat rating of the fuel pin during irradiation was 40 kW/m. After irradiation, the fuel pins of the cluster were subjected to detailed post-irradiation examination using various non-destructive and destructive techniques to assess the performance of the fuel pins. The examination included visual examination, ultrasonic and eddy current testing, gamma scanning, released fission gas analysis and microstructural examination using optical and scanning electron microscopy. No abnormal dimensional changes were observed in the fuel pins, the fission gas release was less than 1% and no fuel restructuring was observed. The results show superior performance of Thoria based fuel as compared to that of UO2 fuel. This report presents the salient observations of the post-irradiation examination on the fuel pins.

Keywords- Thoria, Mixed Oxide, Post irradiation examination, Fuel performance, Cladding.
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Keywords- Thoria, Mixed Oxide, Post irradiation examination, Fuel performance, Cladding.
1. **INTRODUCTION**

India has limited uranium ore, but has vast thorium reserves. For providing energy security on a sustainable basis over a long term, thorium utilisation is the core objective of the Indian Nuclear Power Programme [1, 2]. The third stage of the Indian Nuclear Power Programme is based on the Thorium based fuels. Unlike natural uranium which contains fissile isotope, U$^{235}$, thorium does not contain any fissile material from the uranium cycle [3]. A thorium fuel cycle based Advanced Heavy Water Reactor (AHWR) is being developed for large-scale utilisation of thorium [4]. The reactor is a vertical pressure tube type, heavy water moderated, boiling light water cooled reactor generating 300 MWe. (Th–Pu)$O_2$ and (Th–$^{233}U$)$O_2$ will be used as fuel in the reactor.

Since the available database on irradiation behaviour of the thoria based fuel [5] is limited, research and development in fabrication, characterization and irradiation testing of thoria based fuels has been initiated. In order to study the performance of mixed Thoria-Plutonia fuel during irradiation, short length fuel pins containing sintered pellets of Th$O_2$-4%Pu$O_2$ assembled into a cluster, were irradiated in the pressurized water loop (PWL) of CIRUS reactor up to a burn-up of 18.5 GWd/t [6]. Post irradiation examination (PIE) of the fuel pins from the cluster was carried out at BARC hot cells facility. Non-Destructive Examination of the fuel pins from the cluster included visual examination, fuel pin diameter measurement; leak testing, gamma scanning, gamma spectrometry, ultrasonic testing and eddy current testing. Microstructural characterization on the fuel samples taken from the fuel pins was carried out using optical microscopy, scanning electron microscopy, β-γ autoradiography and α-autoradiography techniques. This report presents the salient observations of the examinations carried out on the irradiated fuel pins.

2. **FUEL PIN FABRICATION**

Short length fuel pins containing (Th-4% Pu)$O_2$ sintered fuel pellets, encapsulated in a free standing Zircaloy-2 cladding tube, were fabricated in Radiometallurgy Division of BARC [7]. The fuel pellets were fabricated using powder metallurgy route. The cladding tube was sealed at either ends with end plugs using tungsten inert gas welding (TIG). The schematic of a single fuel pin is shown in Figure1(a). A fuel pin cluster (AC-6) containing five such (Th-Pu)$O_2$ fuel pins
designated TH-1 to TH-5 and one He filled pin (H-6) was assembled for experimental irradiation in the reactor loop. The Arrangement of fuel pins in AC-6 fuel cluster is shown in Figure 1(b).

Table 1 gives the details of the fuel pins.

Figure 1(a) A typical experimental MOX fuel pin

Figure 1 (b) Arrangement of fuel pins in AC-6 fuel cluster. TH-1 to TH-5 are (Th-Pu)O₂ fuel pins and H-6 is helium filled pin.
Table 1 Details of the Thoria based MOX fuel pins

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<th>Clusters</th>
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<tr>
<td>Clad type</td>
<td>Free standing Zircaloy-2</td>
</tr>
<tr>
<td>Number of pins</td>
<td>6 (5 (Th-4% Pu)O₂ + 1 Helium filled pin)</td>
</tr>
<tr>
<td>PuO₂ content</td>
<td>4%</td>
</tr>
<tr>
<td>Pellet diameter</td>
<td>12.22 ± 0.01 mm</td>
</tr>
<tr>
<td>Pellet length</td>
<td>12.0 ± 1.0 mm</td>
</tr>
<tr>
<td>Pellet density</td>
<td>92-94% TD</td>
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<tr>
<td>Stack length</td>
<td>435 mm</td>
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<tr>
<td>Cladding outer wall diameter</td>
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<tr>
<td>Cladding wall thickness</td>
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</tr>
<tr>
<td>Cold plenum length</td>
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3. IRRADIATION HISTORY

The experimental fuel pin cluster was irradiated in the PWL of CIRUS research reactor. The thermal neutron flux in the loop was 5x10¹³ n/cm²/sec and the temperature and pressure of the coolant in the loop was 240°C and 105kg/cm² respectively. The linear heat rating in the fuel pins was in the range 35 - 40 kW/m. The fuel cluster was irradiated up to a calculated fuel burnup of 18.5 GWd/Te. After irradiation and cooling, the fuel pin cluster was transported to the hot cell facility for post irradiation examination.

4. POST IRRADIATION EXAMINATION

4.1 Visual examination

Visual examination was carried out on the fuel cluster (Figure 2) and individual pins using a wall mounted periscope. All the fuel pins were found having deposit of loose white powder. This may be due to the storage of this fuel cluster in the water pool containing other fuel assemblies with aluminum as the clad. The fuel pins were cleaned by cotton soaked in alcohol to remove the white deposit. No abnormality or defect of any type was visible on the surface of the cladding of the fuel pins. Visual examination of the fuel pins did not show any evidence of corrosion or discoloration of the zircaloy-2 cladding surface.
4.2 Diametral profile

Diametral profile of the fuel pins were measured using a set up fitted with a sliding dial gauge. The diameter was read from the dial gauge display through the periscope. The diameters of the irradiated fuel pins were found to be within the manufacturing tolerances.

4.3 Leak testing

Leak testing was carried out in the hot cell using liquid nitrogen and alcohol leak test method. During this test, each pin was immersed in liquid nitrogen for 5-7 minutes; subsequently the fuel pin was transferred to into a tank containing alcohol. No leaks could be detected, indicating that all the fuel pins were intact.

4.4 Ultrasonic testing

Ultrasonic testing was carried out using two 10 MHz line focused immersion probes fitted at 27° in the probe carriage for detection of axial and circumferential defects. Multi-channel ultrasonic flaw detector was used for slow helical scan combining axial probe translation and rotation of the fuel pin. Figure 3(a) shows the ultrasonic testing set up inside the hot cell and Figure 3(b) shows two channels of ultrasonic flaw detector showing surface signals.

Figure 2 As received AC-6 fuel cluster inside the Hot cell

Figure 3(a) Loading of fuel pin to Ultrasonic scanner using master slave manipulator

Figure 3(b) Two channels of ultrasonic flaw detector showing typical surface signals
4.5 Eddy current testing

Eddy current testing (ECT) was carried out on the fuel pins of the cluster. Figure 4(a) shows the eddy current testing set-up inside the hot cell. ECT of the fuel pins revealed presence of a defect inside the cladding of the fuel pin TH-2. The eddy current signals obtained from the defect location of the fuel pin TH-2 is shown in Figure 4(b). This defect was not open to the surface, as indicated by leak testing. Out of the total five fuel pins, two intact pins (TH-1 and TH-3) were sent for re-irradiation in the reactor loop. Detailed examination was carried out on one intact fuel pin (TH-5) and the defect location of the other fuel pin (TH-2) by destructive testing.

4.6 Gamma scanning

Gamma spectroscopy and gamma scanning using the HPGe detector and multi channel analyzer/scaler (MCA/MCS) were carried out on the fuel pins. Co-60 and Cs-137 sources were used for calibration. The spectra obtained from these elements revealed the presence of Cs-137, Cs-134, Eu-154 and Tl-208. Figure 5(a) shows a typical gamma ray spectrum. Each element was scanned for Cs-137 over the entire length. The dwell time was set to 6 seconds. Gamma scanning over the length of the fuel pin showed higher counts of Cs-137 near the top and bottom portion of the elements probably due to migration of some Cs to these locations which are relatively cooler. The scans showed nearly flat response along the rest of the elements. Figure 5(b) illustrates the scans for TH-1, TH-3 and TH-4 fuel pins.
Figure 5(a) Typical gamma ray spectrum of (Th-4%Pu)O₂ fuel pin

Figure 5(b) Gamma scan of three fuel pins

4.7 Neutron radiography
The fuel pin TH-4 was subjected to neutron radiography in the test reactor CIRUS. The bottom and top end plugs were found to be intact. Multiple cracks in the pellet were observed in the neutron radiograph shown in Figure 6. The plenum spring was also observed to be free from any distortion.

Figure 6 The neutron radiograph of the fuel pin TH-4 shows no abnormality after irradiation

4.8 Fuel pin puncturing and fission gas analysis
Fuel pins were punctured for the measurement of the amount of released fission gases. The volume of the released fission gases and the void volume in the fuel pin were measured to arrive at the pressure of the gas inside the fuel pin. A dual column gas chromatograph and a quadrupole...
mass spectrometer were used to analyze the chemical composition and isotopic composition, respectively, of the collected gases. A detailed description of fission gas release measurement is given in reference [8].

Fission gas analysis was carried out on three fuel pins TH-2, TH-4 and TH-5. The fission gas pressure in the pin TH-4 and TH-5 was 4.4 and 3 atmosphere respectively at ambient temperature and the gases were He, Xe and Kr. The fuel pin TH-2 did not show presence of fission gas inside the pin indicating failure of the fuel; however this could not be detected during leak test. Percentage fission gas release in TH-4 and TH-5 was estimated to be 0.6% and 0.4% respectively.

4.9 Ring tension test (RTT)

Tensile strength was evaluated for fuel pins TH-2, TH-4 and TH-5 using ring tension test method at room temperature using a screw driven universal testing machine. Specimens for ring tension test were in the form of 3.5 mm wide ring without any geometrically reduced gauge section. These rings were cut from the irradiated fuel pin by a slow speed diamond wheel cut-off machine. The test fixture consists essentially of two pieces of semi-circular mandrel, the curvature of which matches with the inner diameter of the ring specimen.

At ambient temperature the ultimate tensile strength (UTS) of the thee clad tubes TH-02, TH-04, TH-05 ranged between 564-648 MPa, 529-598 MPa, 584-876 MPa respectively. The total elongation (TE) for the same tubes ranged between 21-26%, 23-33%, 19-26% respectively. The mean strength and elongation for the three tubes were 608MPa, 23%; 573MPa, 27%; 702MPa, 23% for TH-02, TH-04, TH-05 respectively at room temperature. The specimens from TH-04 were tested at both room temperature and 300°C. At 300°C the mean strength and elongation for TH-04 were 335 MPa and 46% respectively.

4.10 Metallographic examination

The regions of interest in the fuel pin were sectioned using a remotely-operated low-speed precision cutting wheels. The fuel in the cut section was held in place by cold setting resin that is drawn into the fuel by suction from the other end of the fuel pin section. The set section was
further cut, mounted and prepared for metallographic examination using a set of remotely operated grinders and polishers. The prepared section was examined in the as-polished condition using a remotely operated, shielded metallograph. \(\beta\)-\(\gamma\) autoradiography of the metallographic samples were carried out to study the distribution of the fission products (mainly Cs) across the cross section. \(\alpha\)-autoradiography was carried out to analyse the distribution of plutonium in the fuel cross section. Metallographic examination of two fuel pins, TH-2 and TH-5 were carried out.

4.10.1 Cladding defect (Fuel pin TH-2)

Examination of metallographic samples taken from the fuel pin, TH-2, at the defect location revealed presence of a massive hydride blister. Figure 7(a) and (b) show the photomacrograph and \(\beta\)-\(\gamma\) autoradiograph of a section of the pin near the defect in the cladding. The cladding defect in the photomacrograph appeared as cracks on the inner surface of the cladding. A higher \(\beta\)-\(\gamma\) activity (as dark regions) was noticed in the fuel close to region of defect in the cladding as shown in Figure 7(b). Figure 7(c) shows a massive hydride blister formed on the inner surface of the cladding along with cracks in the cladding. The blister was 2.2mm in diameter and 0.64mm in depth. A 150 \(\mu\)m thick hydride layer was observed towards the outer surface of the cladding opposite the hydride blister.
4.10.2 Fuel examination (Fuel pin TH-5)

During fuel pin sectioning, a few intact pellets could slide out from the fuel pin. Figure 8 shows a full pellet from the fuel pin TH-5. The pellet appears to exhibit radial zones: (I) a slightly darker outer rim surrounding the inner zone (II) looking lighter in appearance. The difference in appearance is believed to be due to reduction of porosity in the central portion of the pellet section. The surface of the cut sections were examined through a wall mounted periscope to study the crack pattern from one end of the fuel pin to the other end. Only radial cracks were observed in the fuel sections.
Figures 9(a-c) shows the photomacrograph, β-γ autoradiograph and α-autoradiograph of the fuel sections from the fuel pin TH-5. Macroscopic examination of one of the sections revealed white particles (up to 0.4 mm size) dispersed in the fuel section. These particles had no beta-gamma activity and no alpha activity. Radial cracks were observed in the fuel. No columnar grain formation or grain growth was observed in the fuel. The β-γ autoradiographs revealed an asymmetric distribution of β-γ activity across the fuel cross section. Higher activity was observed in the cracks in the fuel. The α-autoradiograph of the fuel sections revealed a uniform Pu-activity along the cross-section.

Figure 9(a) Photomacrograph, (b) β-γ autoradiograph and (c) α-autoradiograph of fuel section from fuel pin TH-5

Figure 10(a) and (b) show the microstructure at the center and periphery of the fuel section. The porosity was observed to be more at the periphery than at the center of the fuel. Grain pull outs that occurred during sample preparation were observed in the fuel section.

Figure 10 Microstructure at the (a) center and (b) periphery of the fuel section
4.10.3 Cladding oxide layer
Uniform oxide layer was observed on the outer surface of the cladding, whereas a discontinuous oxide layer was noticed on the inner surface. Average oxide layer thickness on the outer and inner surface of the cladding was 1.3 µm and 0.9 µm respectively.

4.10.4 Scanning electron microscopy (SEM)
Samples for SEM examination were prepared using a broken piece of fuel and the fuel particles from the fractured piece were transferred on a replicating tape. Cellulose acetate based replica was prepared from the fractured pieces of the fuel taken from pin. The replica of the fracture surface was coated with silver and examined under SEM. The fuel grain size was measured from the impression of the intergranular fracture of the surface observed on the replica.

Since chemical etching of the Thoria fuel is difficult due to its chemical inertness, grain size and morphology was examined by SEM examination of the fracture surface of fuel. Figure 11(a) shows SEM photomicrograph of the size and morphology of the grains on the replica of the fracture surface of the fuel. The average grain size was found to be 14µm. A bimodal grain size distribution was observed in few regions, with larger grains up to 30µm in size and small grains of 2-5µm size as shown in Figure 11(b). Appearance of submicron size fission gas bubbles was noticed on the faces of smaller grains on the fracture surface (Figure 12 (a) and 12(b)).

![Figure 11(a) Impressions of fractured grains on replicating tape](image1.png)

![Figure 11(b) Grain size distribution the fuel](image2.png)
5. DISCUSSION

Post irradiation examination of five ThO$_2$-4%PuO$_2$ fuel pins of AC-6 cluster irradiated to a burnup of 18.5 GWd/Te show excellent performance of Thoria based fuel during irradiation. None of the fuel pins showed any abnormal corrosion or dimensional changes in the fuel pins and all the pins were intact after irradiation although one fuel pin showed an internal cladding defect due to hydriding. The fission gas release in the fuel pins was found to be negligible and there was no noticeable restructuring of the fuel. The performance of a nuclear fuel is judged by its dimensional stability, the extent of fission gas release and the nature and extent of fuel cladding interaction. In all the three respects, the performance of the fuel pins is found excellent. The results provide confidence for use of this fuel in the forthcoming AHWR.

The fuel pins had experienced a linear heat rating in the range 35-40 kW/m during operation. An estimate of the fuel centre temperature during irradiation was made using computer code PROFESS [9,10] using a temperature dependent fuel thermal conductivity model for ThO$_2$ - UO$_2$ [11] and taking into account the degradation of thermal conductivity with burnup due to fission products accumulation and radiation damage. The maximum fuel centre temperature estimated for the fuel during its irradiation was close to 1100$^\circ$C. This is significantly lower than the temperature expected for UO$_2$ fuel at similar heat rating. Absence of visible fuel restructuring and a very low fission gas release in the fuel pins are in contrast to the usually observed
significant fuel restructuring and appreciable gas release in UO$_2$ fuel irradiated under similar conditions [12]. An UO$_2$ fuel pin in PHWR fuel assembly, which operated with peak linear heat rating of about 400 W/cm up to a burnup of about 12 GWd/t showed presence of a dark porous region containing optically visible fission gas bubbles on the fuel grain boundaries in the central region of the fuel cross section. This microstructure in UO$_2$ fuel indicated the growth and interlinkage of fission gas bubbles on the grain boundaries which is essential for fission gas release to occur from the fuel. The cross section of the ThO$_2$-PuO$_2$ fuel examined in this study did not reveal presence of any visible intergranular gas bubbles during microstructural examination using optical microscopy. This showed that at any given power rating the fission gases can be retained up to much higher burnup in ThO$_2$ based fuel than in UO$_2$ fuel. SEM examination of the fracture surface of fuel pieces showed presence of some submicron size bubbles on the faces of smaller grains. This only indicates that the process of nucleation and growth of fission gas bubbles has started in the smaller grains but the number, size and density of fission gas bubbles was too low to allow an appreciable fractional coverage of grain face area for interlinkage of bubbles to facilitate the release of the gases.

Fission gas release in UO$_2$ fuel depends on fuel temperature and burnup. Vitanza et al [13, 14] has defined an empirical threshold for 1% fission gas release in BWR fuel pins in terms of fuel centre temperature and burnup based on the fission gas release data of fuel pins irradiated in Halden BWR. A similar threshold for 1% fission gas release has also been derived using PROFESS code from theoretical consideration of fission gas release mechanism involving solid state diffusion of gas atoms to the grain boundary [9] and their subsequent release after the grain boundaries are saturated with gas atoms. PROFESS threshold is more conservative than Halden or Vitanza’s threshold. Figure 13 shows the plot representing PROFESS threshold and Halden threshold for 1% FGR. It is found that for a fuel centre temperature of of 1100°C, the fission gas release in the fuel will not exceed 1% up to a fuel burnup of about 19 GWd/t for UO$_2$ fuel.
For ThO₂ based fuel the threshold burnup will be still higher because diffusion coefficient of fission gases in ThO₂ is lower than in UO₂ [15]. If we consider the Halden /Vitanza threshold, then the threshold burnup is still higher. The measured fission gas release of 0.4 – 0.6% in the ThO₂-PuO₂ fuel pins examined in this study is consistent with the above burnup threshold.

Post irradiation examination of CANDU fuel bundles filled with ThO₂-PuO₂ pellets have also been reported to show good performance under irradiation [16, 17]. Thoria based fuel has several advantages over UO₂ fuel, such as higher fuel thermal conductivity over a large range of temperature, its chemical inertness and high melting point. Superior performance of Thoria based fuel over UO₂ fuel is believed to be due to its above properties.
6. CONCLUSIONS

1. Post irradiation examination of five ThO$_2$-4%PuO$_2$ fuel pins of AC-6 cluster irradiated in pressurized water loop of CIRUS up to 18.5GWd/t with peak LHR of 40kW/m was carried out to assess the irradiation performance of fuel chosen for AHWR being developed in India. Four fuel pins remained intact during irradiation. One fuel pin showed an internal hydride defect.

2. PIE observations showed that fission gas release in the fuel pins was negligible (0.4-0.6%), there was no dimensional change in the fuel pins and there was no evidence of fuel restructuring. Good performance of fuel was attributable to lower operating fuel center temperature because of high thermal conductivity of the fuel. The maximum centre temperature of the fuel was estimated to be about 1100°C. The fission gas release in the fuel pin is not expected to exceed 1% up to 20,000MWd/t

7. Acknowledgement

The authors are thankful to Shri D. N. Sah for useful suggestions provided in preparation of the report. Authors would like to express their thanks to Shri Shailesh Katwankar, Shri S.R. Soni and other members of hot cells operation team for their assistance in carrying out PIE of the fuel pins in the hot cells facility.

8. References

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