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THE FISSION GAS BUBBLE DISTRIBUTION IN URANIUM DIOXIDE FROM HIGH TEMPERATURE IRRADIATED SGHWR FUEL PINS

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The fission gas bubble distribution in uranium dioxide from SGHWR fuel pins irradiated in the UKAEA's Winfrith SGHWR has been studied, using transmission and scanning electron microscopy. The fuel pins had been irradiated to a burn-up of ≈1%, at temperatures in the range 400–2300°C and at ratings of 25–46 watt/gram. The fission gas bubbles nucleate heterogeneously. The bubble size increases slightly with temperature and decreases with rating. The weak temperature dependence of the bubble size indicates an irradiation enhanced gas atom diffusion coefficient. There is no evidence of appreciable gas bubble mobility. The fraction of gas produced which is contained in the bubbles has a maximum value at ≈1650°C. Except in the restructuring regime the dominant mechanism leading to the release of gas by its transfer to grain boundaries is the diffusion of single atoms. This applies at all irradiation temperatures and implies that models of gas release developed for lower temperature AGR fuel can be applied to gas release in SGHWR fuel pins.

La distribution des bulles de gaz de fission dans du bioxyde d'uranium provenant d'aiguilles de combustible du réacteur SGHWR irradiées dans le réacteur SGHWR Winfrith de l'UKAEA a été étudiée en utilisant la microscopie électronique par transmission et à balayage. Les aiguilles de combustible ont été irradiées à un taux de combustion de ~1%, à des températures situées entre 400 et 2300°C et à des taux de 25–46 watt/gram. La germination des bulles de gaz de fission s'est révélée hétérogène. La taille des bulles augmente légèrement avec la température et diminue avec le taux d'irradiation. Le fait que la taille des bulles dépende peu de la température indique que le coefficient de diffusion des atomes gazeux est augmenté par l'irradiation. Par contre, il n'y a pas d'évidence d'une mobilité appréciable des bulles de gaz. La fraction de gaz produits qui est continue dans les bulles présente une valeur maximale à ≈ 1650°C. Sauf dans le domaine de restructuration le mécanisme prédominant conduisant au dégagement de gaz par son transfert aux joints de grains est la diffusion des atomes un à un. Ceci s'applique à toutes les températures d'irradiation et implique que les modèles de dégagement de gaz pour le combustible AGR à plus basse température puissent être appliqués au dégagement de gaz dans les aiguilles de combustible SGWHR.

Die Spaltgasblasenverteilung in Urandioxid aus SGHWR-Brennstäben, die im SGHWR Winfrith der UKAEA bestrahlt worden waren, wurde transmissions- und rasterelektronenmikroskopisch untersucht. Die Brennstäbe waren bis zu einem Abbrand von etwa 1% zwischen 400 und 2300°C sowie bei einer spezifischen Leistung zwischen 25 und 46 W/g bestrahlt worden. Die Keimbildung der Spaltgasblasen erfolgt heterogen. Die Blasengrösse steigt geringfügig mit der Temperatur an und fällt mit der spezifischen Leistung. Die schwache Temperaturabhängigkeit der Blasengrösse weist auf einen bestrahlungsinduzierten Diffusionskoeffizienten für Gasatome hin. Es gibt keine Anzeichen für eine nennenswerte Beweglichkeit der Gasblasen. Der Anteil des gebildeten Gases, das sich in den Blasen befindet, hat einen Maximalwert bei etwa 1650°C. Die Restrukturierungsphase ausgenommen, besteht der vorherrschende Mechanismus, der zur Gasfreisetzung durch Transport zu den Korngrenzen führt, aus der Diffusion einzelner Atome. Das gilt für alle Bestrahlungstemperaturen und besagt, dass die Gasfreisetzungsmodelle, die für AGR-Brennstoffe mit niedrigeren Temperaturen aufgestellt wurden, auf die Gasfreisetzung in SGHWR-Brennstäben angewendet werden können.

1. Introduction

Cornell [1] has characterised the intragranular fission gas bubble distribution in uranium dioxide from Windscale AGR fuel pins irradiated at temperatures ⩽1300°C to ≈1% burn up and at ⩽1600°C to ≈0.1% burn up. Some SGHWR fuel pins will experience centre temperatures ⩾1600°C where the population of intragranular gas bubbles has not been studied previously. The present paper reports the extension of observations into this temperature range.

Fission of U_{235} produces 25 stable rare gas atoms

for every 100 atoms consumed. These gas atoms are insoluble in uranium dioxide and precipitate as inter and intra-granular bubbles. The size, distribution and stability of these bubbles decide the extent of fuel swelling and gas release.

The gas residing as intragranular bubbles might be expected to reduce the accumulation of gas at grain boundaries, however, an irradiation induced re-solution process acts on all bubbles [2,3]. The gas bubbles are nucleated heterogeneously along the fission fragment tracks [4] and they grow by collecting gas atoms and vacancies. The growth time is, however, short before the bubble is completely re-dissolved in a single event by interaction with a fission fragment. This irradiation re-solution limits the size to which the intragranular bubbles grow (≈3 nm diameter at 1300°C).

The gas can move to the grain boundaries as single atoms via multiple fission induced re-solution and subsequent atomic diffusion [5]. As the temperature is increased in the range 1000–1600°C an appreciable fraction of the gas will reside as large intergranular bubbles [6]. It is the inter-linking of these grain boundary bubbles which releases gas into the plenum.

Above 1600–1700°C the uranium dioxide re-structures and columnar grains form [7]. In the columnar restructuring range the bubbles can move by a vapour transport mechanism up the temperature gradient and gas release is high.

The present study describes an examination of the fission gas bubble distribution in uranium dioxide from experimental SGHWR fuel pins irradiated in the UKAEA's Winfrith SGHWR to ≈1% burn-up at temperatures ≤2300°C and at ratings of 25–46 watt/gram. The fuel pins exhibit both equiaxed and columnar crystal grains.

2. Experimental

The fuel pins examined were taken from experimental SGHWR clusters. The uranium dioxide pellets in the pins were 14.3 or 14.5 mm diameter, 15 mm long with both ends dished to a depth of 0.4 mm and with a density ≈97% theoretical. The effect of the reduced diameter was to increase the fuel-clad gap and hence increase the pellet temperature. The pellets in one pin (No. 4) had a higher enrichment of U_{235} and hence higher ratings.

Failure occurred in one of the pins (No. 3) after 206 effective full power days and the cluster was discharged 98 e.f.p.d. later. Over the course of the irradiations the reactor was subjected to periods of daily power cycling during which the power was reduced to 70% for ≈4 hours.

Table 1 lists the mean, discharge and maximum ratings, surface and centre fuel temperatures and enrichments etc. for the pins examined and fig. 1 shows macro-photographs of transverse sections of the pins.

The fuel temperatures quoted in table 1 need some explanation. The temperatures were calculated using computer programmes with attempts to compensate for changes in the fuel clad gap and gas conductivity with the increase of released fission gas. In pins 1 and 4 the fuel clad gap was small and the compensating factor also small. The fuel temperature only varied with rating and the computer calculated discharge temperatures are used. In pin 2 the fuel clad gap was large and the computer calculated centre temperature at discharge was ≈2000°C, which would indicate some columnar restructuring at the centre. Because this restructuring was not observed the temperatures used for pin 2 are the calculated mean fuel temperatures during irradiation.

Table 1

Pin number	% Enrichment	Sample burn-up fissions/m^3	Sample rating watt/gram			Centre temperature °C		Surface temperature at discharge °C
			Peak	Mean	Discharge	Mean	Discharge	
1	1.24	2.0×10^{26}	29	28	27	1360	1335	410
2	1.24	2.0×10^{26}	29	28	27	1830	2000	830
3	1.24	1.9×10^{26}	27	26	25	1765	2300 *	900 *
4	4.3	3.55×10^{26}	46	35	33	1880	1800	535

* Estimated.

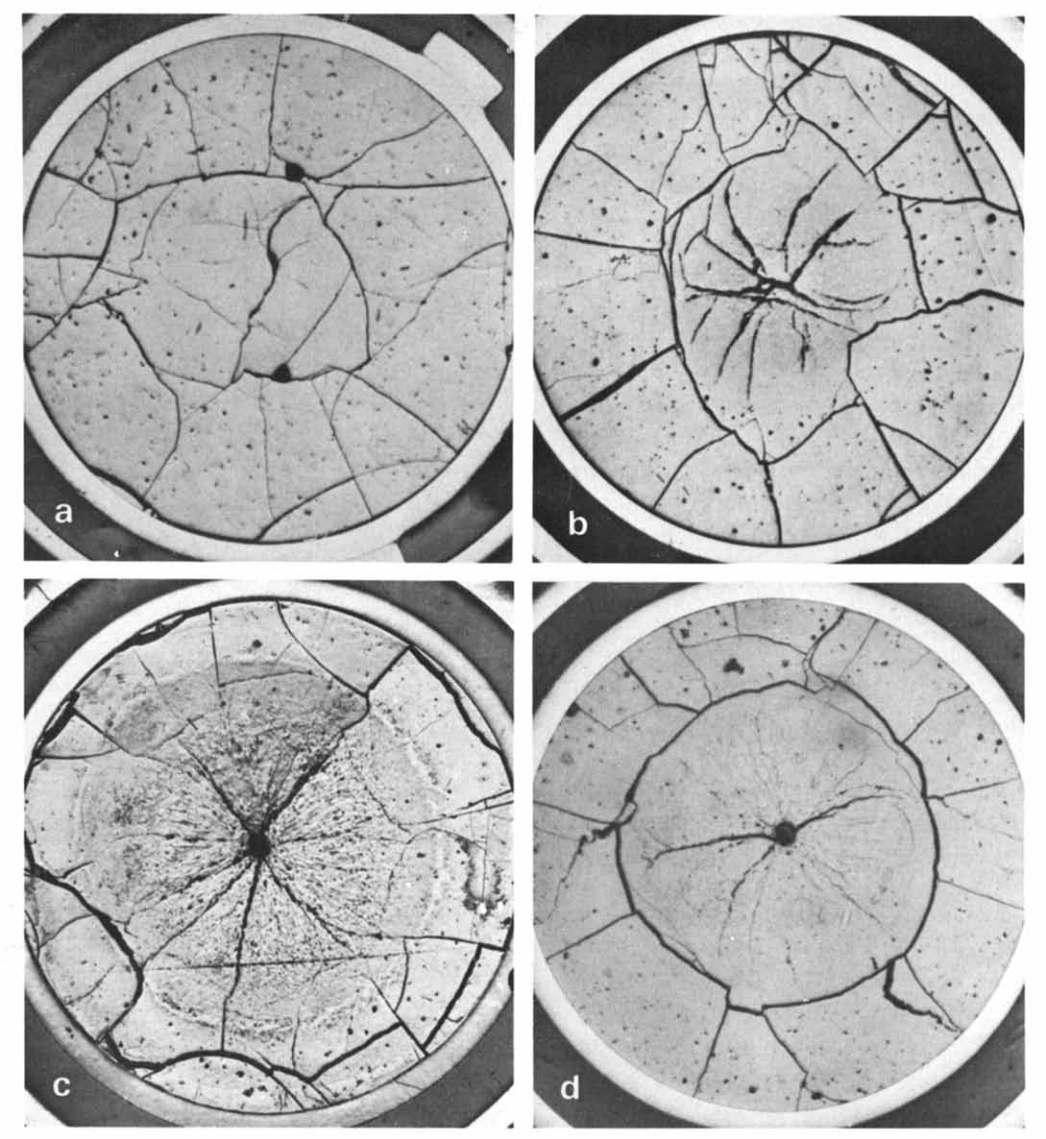

Fig. 1. Optical micrographs of transverse sections of the SGHWR fuel pins examined (a) pin 1 (b) pin 2 (c) pin 3 and (d) pin 4. The fuel pin diameter is 14.5 mm.

Optical microscope examination of the failed pin (No. 3) indicated columnar grains over the central half of the radius and hence a higher temperature than that expected from calculations. This higher temperature would probably be due to a combination of insulating deposits on the clad and changes in the fuel-clad gap. It is possible to assign more likely centre and surface temperatures to the pin by noting the radial positions at which certain specified changes in the microstructure occur, e.g. start of grain boundary porosity, onset of grain growth and columnar restructuring and by comparison with micro-structures from other pins. From this comparison the centre and surface temperatures at discharge have most probable values of 2300 and 900°C.

Transverse sections (≈1 mm thick) were cut from

the fuel pins using a diamond slitting wheel and these were ground on both sides to a thickness of ≈0.25 mm and polished on one face to a 1 μm diamond finish. These sections were fractured along the cracks which arise during irradiation and by suitable selection of the pieces it was possible to designate the area from which they came and from the temperature profile across the pin to assign an irradiation temperature to them.

The small pieces were electrolytically polished to produce thin foils using a "UNITHIN" twin jet polishing machine mounted in a shielded glove box [8]. The thin foils were examined in a 100 kV transmission electron microscope and, after coating with aluminium, in a scanning electron microscope.

No additional shielding of the specimen chambers of the electron microscopes was necessary and maximum irradiation levels at the operator due to the specimens were ⩽0.2 mR/hour.

3. Results

The effect of temperature on the fission gas bubble distribution was best seen in pins 1 and 2 which had centre temperatures of 1360 and 1830°C respectively. Thin foils over the temperature range 900–1830°C showed a high density of small intragranular gas bubbles. These bubbles were best seen in the under or over-focussed condition when they appeared light or dark respectively. The bubbles formed in lines and this was particularly noticeable above 1000°C. As the temperature increased these lines became slightly longer and more clearly defined. Examination of stereo-pairs of micrographs confirmed that the lines of bubbles were straight and in many cases traversed the foil thickness. The directions of the lines were plotted on stereographic projections and were totally random. Evidence for the nucleation of all the bubbles in any particular line at the same time was occasionally observed in that the line was not a discrete line of bubbles but more like a decorated dislocation, e.g. fig. 2. The bubbles often had a square image with the sides of the square parallel with {110} fig. 3.

Up to 1700°C the gas bubble distribution was fairly uniform but above 1700°C the bubble density increased near grain boundaries and the distribution became non-uniform within the grains. Inside the grains there were areas devoid of bubbles and there was a

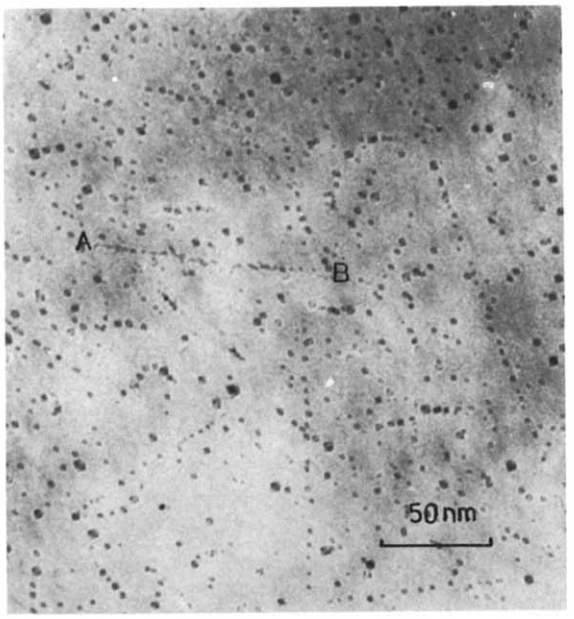

Fig. 2. Transmission electron micrograph taken over-focus of a thin foil from pin 2 at an irradiation temperature of 1650°C. The line marked AB is seemingly just forming and looks more like a decorated dislocation than a line of bubbles.

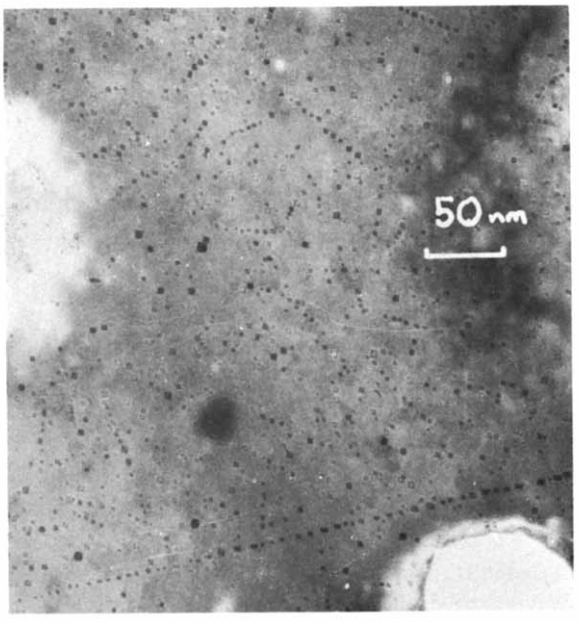

Fig. 3. Transmission electron micrograph taken over-focus of a thin foil from pin 2 at an irradiation temperature of 1800°C. The square sides of the bubble images are parallel with {110}.

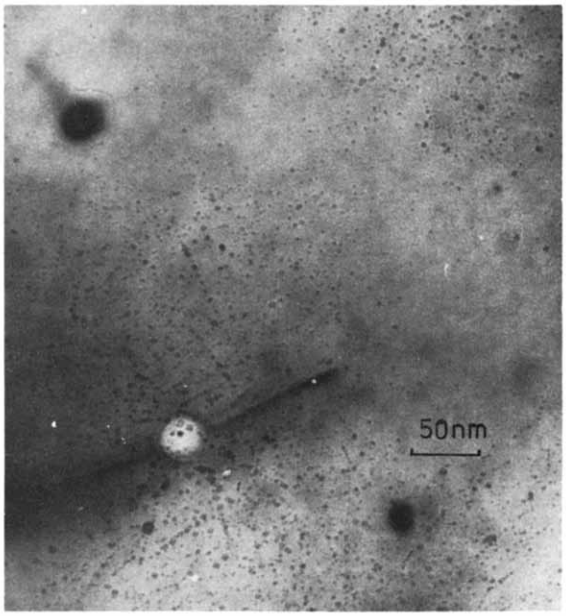

Fig. 4. Transmission electron micrograph taken over-focus of a thin foil from pin 2 at an irradiation temperature of 1800°C. The gas bubble distribution is non-uniform with a higher concentration near the large gas bubbles on dislocations but with other large areas denuded of bubbles.

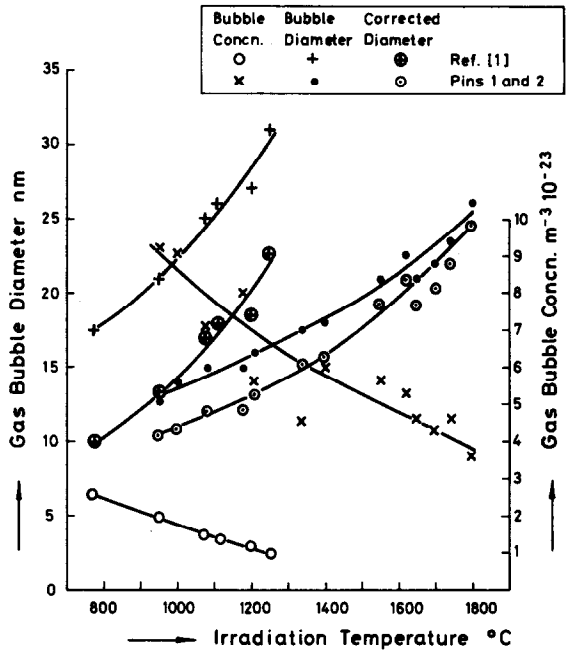

Fig. 5. The variation of the intra-granular fission gas bubble diameter and concentration with irradiation temperature for pins 1 and 2 and also an AGR fuel pin [1].

higher density of bubbles in the immediate vicinity of the large bubbles which nucleated on dislocations and on sub-boundaries, fig. 4. This tendency to nucleate bubbles on dislocations increased with temperature.

Scanning electron microscopy of the polished and thinned foils confirmed that the grains remained equiaxed to temperatures of ≈1600°C and above this they showed a slight tendency to elongate in the direction of the temperature gradient. The grain boundary porosity elongated in the same direction and was extensively inter-linked. Above 1750°C the grains frequently exhibited internal sub-grains with networks of bubbles along the sub-boundaries.

To measure the bubble size it is first necessary to consider the contrast of small bubbles. Rühle [9] has shown that the most accurate value of the diameter is obtained in the under-focussed condition from the measurement of the inner diameter of the first dark fringe. However, even this measurement will result in over-estimates of the diameter by ≈10% at 2 nm diameter and ≈60% at 1 nm diameter.

Other errors can arise in the measurement of the bubble diameters in that the sharpness of the image for these small bubbles is dependent on the foil thickness and the condition of the microscope and the bubbles often have a square or irregular image.

The thicknesses of a number of foils exhibiting sharp bubble images were calculated from parallax measurements on stereo-pairs of micrographs and these were found to be in the range 15–35 nm with a mean value of 25 nm. This value was used in the calculation of bubble densities. Foils with thickness above 35 nm were transparent to the electron beam but with reduced resolution of the small bubbles.

The variation of the as measured intra-granular gas bubble size and density with the irradiation temperature for pins 1 and 2 and for a Windscale AGR pin [1] is shown in fig. 5. The bubble size increased and the bubble density decreased over the temperature range 900–1800°C. The bubble density was higher than that measured by Cornell [1] and this is explainable on the improved foil preparation technique in the present study which resulted in thinner, more uniform foils. Cornell assumed a foil thickness of 150 nm. The bub-

ble diameters in the present study were smaller than those measured by Cornell [1] and this is explainable because Cornell measured to the centre of the first dark fringe which is known to result in an overestimate. When the as-measured diameters in the present study and those on the WAGR pin are corrected following Rühle [9], the bubble diameters in the WAGR pins are still larger than in the SGHWR pins but the difference is not as great as before correction, fig. 5.

The number of gas atoms contained in the intragranular bubbles has been calculated from the bubble diameters and densities. The diameters used are the Rühle corrected values and the bubble densities above 1700°C are corrected for the denuded areas. There are still likely to be small errors in these gas atom content measurements because of thickness and diameter measurements which will probably tend to over estimate the gas content. The variation of the gas content of the bubbles with irradiation temperature is shown in fig. 6. The gas content is plotted as the percentage of the total fission gas produced which is contained in the intra-granular bubbles. The gas content is ≈20% of that produced up to 1300°C with an increase above this to a peak of ≈40% at 1650°C. Above 1650°C there is some evidence that the gas content of the bubbles is decreasing.

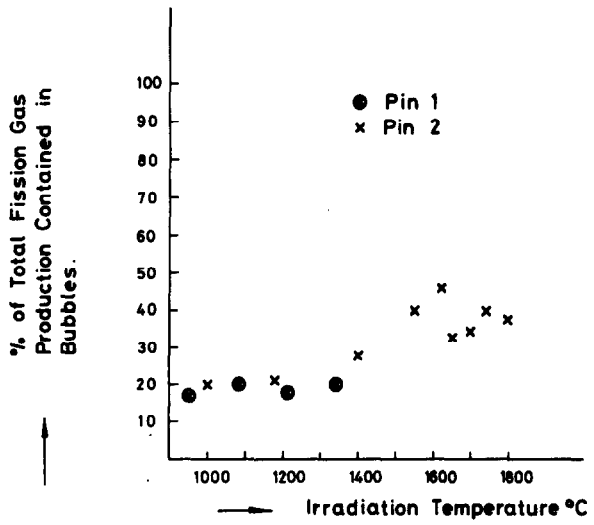

Fig. 6. The variation with irradiation temperature of the percentage of the total fission gas production which is contained in the intra-granular gas bubble distribution.

To study uranium dioxide irradiated above 1800°C pin 3 has been examined. The central 3.5 mm of this pin exhibited a columnar structure and scanning electron microscopy of foils from this region showed large elongated grains with grain boundary bubbles joining together to form tunnels parallel with the temperature gradient.

At irradiation temperatures of 1200–1600°C the intragranular bubbles exhibited a different distribution from that in pins 1 and 2. Although the bubble size was comparable, the bubble density was three times lower and there was little evidence for the formation of lines of bubbles, fig. 7. The bubble density increased in the immediate vicinity of the grain boundary bubbles and as the temperature increased the density near these large bubbles increased. At temperatures above 1700°C the bubbles in the grains became fewer in number until at 2000°C bubbles were only observed within the immediate vicinity of the grain boundary bubbles.

The other difference observed in pin 3 was that the dislocations were heavily decorated with bubbles and solid fission product precipitates.

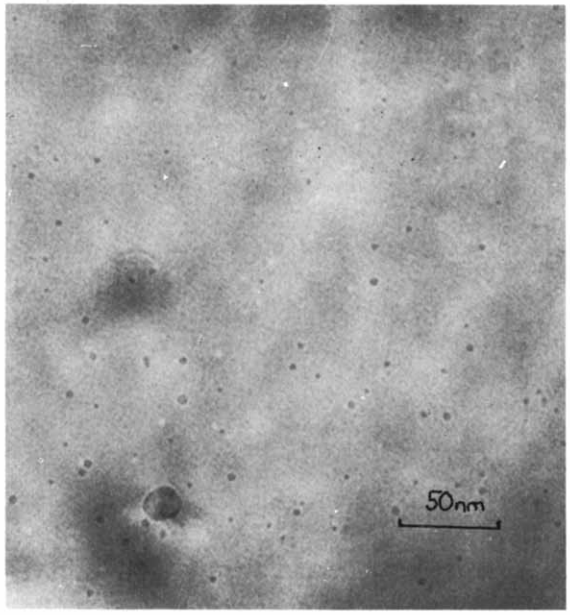

Fig. 7. Transmission electron micrograph taken over-focus of a thin foil from pin 3 at an irradiation temperature of 1560°C. The gas bubbles are at a lower density than in pins 1 and 2 and the tendency to nucleate in lines is not apparent.

At temperatures above 2000°C the thin foils contained a second phase which was almost certainly a reaction product from the ingress of coolant into the failed pin. This second phase was in the form of a diffuse precipitate ≈100 nm in size exhibiting weak strain contrast.

The effect of rating was studied on pin 4. This pin had a fuel enrichment of 4.3% and a high time-averaged rating of ≈35 watt/gram. The maximum rating of ≈46 watt/gram was experienced early in life with a rating over the last period of ≈33 watt/gram.

When thin foils from the edge of this pin were examined the gas bubbles were barely visible up to 1000°C. Even at 1400°C the bubbles were still very small and they did not nucleate in straight lines but rather in short wavy lines, fig. 8. Examination of stereo-pairs of micrographs indicated that some bubble lines were apparently branched and took the form of a letter "Y". The individual bubbles along the tracks were often difficult to resolve, the tracks looking more like lines of inter-connected bubbles. At higher irradiation temperatures, the bubbles were still small with a mean diameter of only ≈1 nm i.e. of the same order as the point resolution of the electron microscope at 100 kV. There was a greater tendency at these temperatures for the bubbles to form in straight lines. The bubble concentration was in excess of 3×10^{24} m^{-3} but not all the bubbles in the tracks were resolvable. The amount of gas in the bubbles was ⩽20% of that calculated from the burn-up, however, the possible errors in this value are considerable.

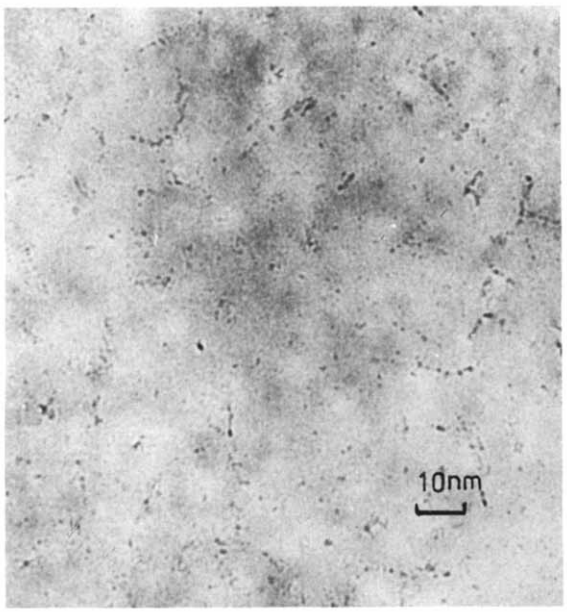

Fig. 8. Transmission electron micrograph taken over-focus of a thin foil from pin 4 at an irradiation temperature of 1350°C.

At higher temperatures the grains developed an internal fine sub-grain structure and these sub-grain boundaries and any disolcations present were decorated with large bubbles.

4. Discussion

The study has shown that intra-granular fission gas bubbles are observed in irradiated uranium dioxide over a wide range of temperature, temperature gradient and rating. The experimental results seem to confirm that the bubbles nucleated heterogeneously along fission fragment tracks [1]. The density of fission tracks expected to be visible at these fission rates, and assuming that the re-solution parameter is as determined by Turnbull and Cornell [10], is $\approx 10^{14}$ mm^{-3}. The measured density of bubble delineated tracks in pin 2 at 1500°C is $\approx 5 \times 10^{13}$ mm^{-3}, although this may be open to the error of counting short lengths. Consequently, the bubble line density is in reasonable agreement with the expected surviving fission track density.

Evidence was obtained for in-situ nucleation and growth of the bubbles on the fission tracks whilst there was no evidence of bubble movement onto the tracks. At the higher irradiation temperatures the bubble tracks became longer and more clearly defined and nearly all the bubbles could be established as lying on lines.

Bubble densities and also fission track densities were much higher and bubble sizes smaller at higher ratings and the fission gas bubble tracks were short and wavy. This higher density and smaller size is most likely to be a consequence of the larger re-solution parameter at higher ratings. Pati, Dapht and O'Boyle [11] have reported calculations showing that the bubble size is reduced with increasing re-solution parameter. These authors, however, assume a constant number of bubble nuclei. Speight [12] has shown how, assuming the bubble is destroyed by a single encounter with a fission fragment and the bubble nucleation size

is negligibly small, it is possible to calculate the spread in bubble size. The distribution can be plotted for the same temperature but different ratings and assuming that the rating does not significantly alter the gas atom diffusion coefficient. It is found that the mean bubble size of the distribution at 1400°C increases by ≈15% when the rating is decreased from 35 to 25 watt per gram. Thus there is expected to be an effect of rating on bubble size although the variation observed experimentally is much greater than calculated.

It is likely that at least part of the discrepancy in the corrected bubble size and density between the results of Cornell [1] and those of the present study is because the WAGR ratings were 10-20 watt/gram whereas the SGHWR ratings were ⩾25 wtt/gram.

The only pin in which the linear nucleation of bubbles was not readily apparent was pin 3. The bubbles in this pin exhibited a size against irradiation temperature variation comparable to that found in other equivalently rated pins, but the bubble density was a factor 3 down. A consequence of the high centre temperature in pin 3 was that temperature gradients were higher than in the other pins. Unfortunately it is not known whether the ingress of coolant has affected the fission gas bubble distribution. The build-up of bubbles near grain boundaries in pin 3 seems to indicate re-solution of gas from grain boundary bubbles.

In pin 2 the intra-granular gas bubble distribution became inhomogeneous at the higher irradiation temperatures, i.e. ⩾1700°C. There were areas ≈2 μm wide denuded of gas bubbles and higher concentrations were found in the immediate vicinity of the larger gas bubbles on dislocations and sub-grain boundaries. Thus the ease of formation of stable gas bubble nuclei seems to fall off with increased temperature.

Considering now the growth of the intra-granular gas bubbles. Cornell [1] found only a weak dependence of size on irradiation temperature and this has been confirmed by the present work. Turnbull [4] explained Cornell's results by proposing that the bubbles grow for a time that is short compared with the irradiation time. The bubble is then knocked back into solution by a single fission fragment. It is only the growth phase that depends on the irradiation temperature, via the gas atom diffusion coefficient D, and the irradiation dose, via the gas atom concentration C. The bubble radius is then given by

$$R^4 = R_o^4 + 2bCD/\pi F\lambda , \qquad (1)$$

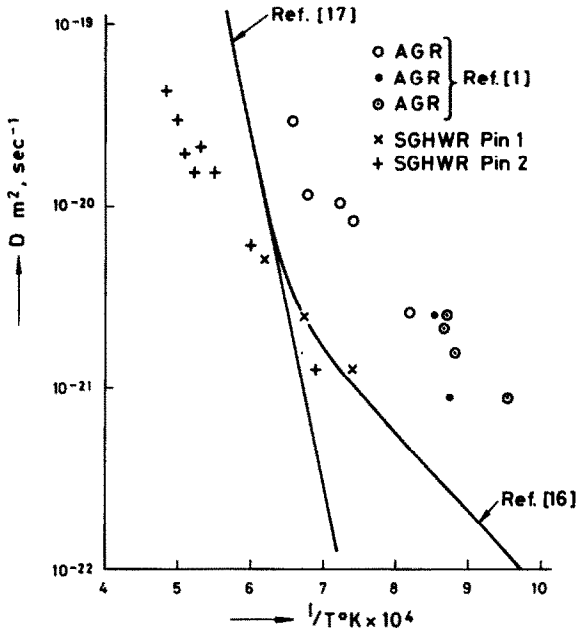

Fig. 9. The variation of the gas atom diffusion coefficient D, calculated from the bubble radius, with temperature for pins 1 and 2. Also plotted are the results of Cornell [1] and [17] and the calculated curve of Speight [16].

where R_o is the bubble nucleus radius assumed to be 0.5 nm, b = Van der Waal's constant, F = fission rate per unit volume and λ is the fission fragment range (≈6 μm, Noggle and Stiegler, [13]).

The weak dependence of the bubble size on the operating conditions is obvious from eq. (1).

Other theories have proposed that the bubble nucleation is homogeneous [14,15]. However, Turnbull's model [4] is perhaps the one which more readily explains the electron microscope observations.

The corrected values of R have been used to calculate the gas atom diffusion coefficient at the various irradiation temperatures and the results are shown in fig. 9. The results for pin 3 have not been included because of the variability of size near grain boundaries nor the values for pin 4 because in this pin the bubble radius over the whole temperature range was of a size comparable with R_o. Also plotted are the results calculated from Cornell [1], the irradiation enhanced diffusion coefficient calculated by Speight [16] and the values calculated by Cornell [17] from post irradiation annealing studies. Considering the inaccuracies in the measurement of R and its 4th power dependence the

fit of the measured values of D with the calculated values of Speight [16] is good.

The bubble concentration is zero at the start of irradiation but soon reaches an equilibrium concentration C_1, when the rate of creation is balanced by the re-solution rate. Turnbull [4] has shown that when equilibrium is attained,

$$C_1R^2 = \alpha/\pi\lambda\,, \qquad (2)$$

where α is the number of bubbles nucleated per fission fragment. The results of Cornell [1] indicate that C_1R^2 is approximately constant and that α has a value of ≈ 5. In the present study C_1R^2 is again approximately constant and $\alpha \approx 10$. This is comparable with the number of bubbles per line at lower temperatures but too low at higher temperatures, e.g. at 1800°C the number of bubbles in one line has been observed to be in excess of 100.

The results of the present study have shown that the intra-granular fission gas bubbles remain small up to temperatures of ≈2000°C. The only large intra-granular bubbles observed are those on dislocations and sub-boundaries. Models developed to account for the fission gas behaviour below 1300°C are applicable to higher irradiation temperatures.

In the temperature range 1600–1700°C there is a maximum amount of gas as intra and inter-granular bubbles. The fission gas content of the grains decreases above 1700°C. This is consistent with the well documented measurements of high gas release for pins operating above 1600°C.

5. Conclusions

1) Intra-granular fission gas bubbles in uranium dioxide, irradiated to ≈1% burn-up at 25-33 watt/gram nucleate heterogeneously. At temperatures up to 1800°C the main nucleating sites are along fission fragment tracks. Above 1800°C the bubbles nucleate on dislocations and sub-boundaries and in the immediate vicinity of grain boundary bubbles.

2) The bubble size increases slightly and the bubble density decreases slightly with increasing irradiation temperature.

3) Increasing the rating leads to higher bubble and bubble line densities and smaller bubble sizes.

4) There is no evidence for the mobility of intra-granular bubbles.

5) The fraction of the fission gas produced which is contained in the intra-granular bubbles has a maximum value of ≈40% at 1650°C.

6) The weak temperature dependence of the intra-granular fission gas bubble diameter indicates a temperature insensitive, and therefore irradiation enhanced, gas atom diffusion coefficient.

7) Except where migrating grain boundaries form columnar grains, the dominant mechanism leading to the release of gas by its transfer to grain boundaries is the diffusion of single atoms. This applies at all irradiation temperatures and implies that models of gas release developed for lower temperature AGR fuel can be applied to calculate gas release in SGHWR fuel pins.

Acknowledgements

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