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**AEC Research and
Development Report**

**Knolls
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**Metallography of
Irradiated
UO₂-Containing
Fuel Elements**

W. K. Barney

B. D. Wemple

January 15, 1958

**Operated for the
United States Atomic
Energy Commission by**

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UC-25, Metallurgy and Ceramics
(T1D-4500, 13th ed)

General Electric Company
KNOLLS ATOMIC POWER LABORATORY
Schenectady, New York

METALLOGRAPHY OF IRRADIATED UO_2 -CONTAINING FUEL ELEMENTS

W. K. Barney
B. D. Wemple

January 15, 1958

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Authorized Classifier

2/11/58
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ABSTRACT

A description of hot laboratory metallography and techniques of operation are presented. These facilities and techniques provided a means of examining fuel elements that contain UO_2 after irradiation to high burnups. Some unusual irradiation characteristics of UO_2 were observed and each effect is discussed. A complete explanation of the causes of such effects has not yet been obtained.

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METALLOGRAPHY OF IRRADIATED UO_2 -CONTAINING FUEL ELEMENTS

W. K. Barney, B. D. Wemple

INTRODUCTION

Metallography proved especially useful in the study of the behavior of fuel elements under irradiation. The use of metallography for examination of radioactive specimens is considerably more complicated than for examination of ordinary materials, but improvements in hot laboratory techniques are making it possible to obtain much valuable information. This report describes the KAPL procedure for performing remote metallography and discusses the observations of irradiated fuel elements.

DESCRIPTION OF ELEMENTS

Data were obtained on two general types of elements, the stainless steel dispersion type and the annular oxide type. Both types of elements consist of enriched UO_2 confined by stainless steel but the theory behind each element is decidedly different. The UO_2 of the annular element is a low-density layer formed by steam oxidation. The dispersion elements contained high-density, high-fired UO_2 particles. As demonstrated, this leads to considerable difference in the resulting UO_2 after irradiation.

METALLOGRAPHIC EQUIPMENT AND PROCEDURE

The samples were processed in a 4-by-14-ft cell with 9-in.-thick steel walls. Six Central Research Laboratory Model No. 4 Argonne type slaves were used for remote manipulation of the metallographic equipment in the cell.

Figure 1 is a diagram of the cell and equipment and Figure 2 is a general view of the front of the metallographic cell showing the equipment used to process the samples.

Mounting of Samples

The samples were mounted in Melmac powder by means of a Precision Jarrett Aeromount Mounting Press. Spring clips were used to hold the samples in the desired position during mounting. The samples to be processed were approximately $1/4$ to $3/8$ in. in length. Figure 3 shows the Precision Jarrett Mounting Press.

Grinding

The samples were ground by means of three Buehler No. 1505-2F AB low-speed polishers equipped with Buehler No. 1900-AB Automet Polishing attachments, shown in Figure 3. Automet silicon carbide pressure-sensitive grinding paper (No. 1467 SW), with adhesive backing, was used for grinding the samples. Figures 3 and 4 show the 5-hole specimen holder and specimen loading assembly used with the Automet polishing attachments. The attachments made it possible to process five samples at one time and apply up to 60 lb pressure on the samples during grinding operations.

The samples were ground on 240-grit paper for approximately 7 to 8 min with an initial pressure of 50 lb applied to the samples. After approximately 4 min of grinding at 50 lb, the pressure was gradually decreased to approximately 10 lb. The samples were ground on 400-grit paper for approximately 5 min, starting with an initial pressure of 40 lb. After 3 min of grinding, the pressure was gradually reduced to 10 lb. Finally, the samples were ground on 600-grit paper for 5 min, starting with an initial pressure of 30 lb. After 2 min, the pressure was gradually reduced to 5 lb. Water was used as the coolant for all the grinding operations. The samples and the holder were cleaned with cotton and water after completion of the grinding operations.

Lighter pressures and less grinding time were used while processing the annularly loaded samples. These were ground on 240-grit paper for 5 min, starting with an initial pressure of 40 lb, on 400-grit paper for 5 min with an initial pressure of 30 lb, and on 600-grit paper for 4 min with an initial pressure of 20 lb. The lighter pressures made possible better retention of the UO_2 in the samples.

Polishing

The samples were polished by means of two Buehler No. 1505-2F AB low-speed polishers equipped with Buehler No. 1900-AB Automet polishing attachments (See Figure 3). One unit was used for coarse polishing and the other for fine polishing.

The samples were coarse polished on Airplane wing cloth (Air Associates, Inc.) with Precisionite polishing powder, composed of 0- to 3-micron particles of aluminum oxide. The coarse polishing time was 8 to 10 min at 5 lb pressure.

Fine polishing was accomplished on Gamal cloth (Fisher Scientific Company) or Suede cloth (Phillips Premier Company) with Precisionite polishing powder, composed of 0- to 0.1-micron particles of aluminum oxide. The fine-polishing time was 6 to 7 min at 5 lb pressure.

Water was generally used as the coolant during polishing. However, to reduce the amount of cold-work produced on the samples during polishing, a 10% solution of oxalic acid in water was occasionally applied to the polishing wheels during the polishing operations.

The samples were cleaned with cotton swabs and water after polishing. A hot-air blower was used to dry the samples as shown in Figure 4.

Etching

Aqua regia (10 parts nitric acid, 20 parts hydrochloric acid, and 30 parts Ethylene Glycol) was used as the etchant. The samples were etched by swabbing with cotton dipped in the etchant solution. The etchant fumes were removed from the cell by placing the etching solution under a vacuum hood as shown in Figure 4. After etching, the samples were cleaned with cotton and water and dried by means of the hot-air blower.

Occasionally, it was found necessary to repolish and re-etch the samples to remove all the cold-work that occurred during the grinding and polishing operations. The samples were repolished on the fine polishing wheel for approximately 3 min at 3 to 5 lb pressure and then re-etched.

Examination and Photomicrographs

The examination and photomicrographing of the samples were accomplished with a shielded Bausch and Lomb Research Metallograph located outside the cell, as shown in Figure 1. The samples were transferred from the interior of the cell to the stage of the metallograph with a Castle Manipulator. Remote controls were extended through the shielding of the metallograph to operate the various movements of the stage. Figure 5 is a general view of the metallograph and its auxiliary equipment.

The samples were examined and photomicrographed at magnifications varying from 25X to 1000X in the "as polished" and "as etched" conditions. Bright field illumination was used for examining and photographing the samples.

Results

Excellent retention of the UO_2 particles in the dispersion and annular loaded elements was obtained. Also, the metallographic procedure produced a very good surface for examining and photographing the samples in the "as polished" and "as etched" conditions.

With the metallographic equipment and procedures described, it was possible to process five samples for examination in the "as polished" condition in approximately 8 man-hours. An additional 4 man-hours were required to etch, examine, and photograph each sample.

More detailed information on the equipment is given in the Fifth Hot Laboratories and Equipment Conference Manual.¹

OBSERVED IRRADIATION EFFECTS

The object of the metallographic study of the two types of fuel elements was to determine what changes had occurred during irradiation. The over-all quality of the irradiated element can be determined by visual and dimensional examination and mechanical property determinations. These tests may give an adequate picture of the behavior of a particular element but little is told about its future capabilities, nor is its behavior explained. Metallographic examination of the elements was performed to obtain the data required to answer these questions and to validate the design theory of the elements.

The microstructure of the various elements, which operated at widely varying conditions, is described in this section. The UO_2 is of primary interest and most of the discussion centers about the observed effects of irradiation on this phase. The stainless steel is not significantly affected except for metal that is damaged or affected by recoiling fission fragments, but this is also described in detail. The significance of the observed effects on element performance is discussed in each section.

Sintering of UO_2

The maximum internal temperature of the annular oxide element was calculated at approximately 1600°C , assuming one atmosphere of helium and no sintering of the loose UO_2 powders. It has been shown that the UO_2 does not remain a powder for any appreciable length of time because all the elements that were metallographically examined were found to have undergone marked sintering. Elements with 4% fission of the U^{235} showed that the UO_2 was a solid mass with no evidence of powders remaining. (See Figure 6.) The maximum temperature reached in this type of element has not been measured but it is believed that it reaches its maximum value immediately after insertion and drops quite rapidly as the UO_2 powders sinter. It is certain that the UO_2 did not become molten and what was observed was a sintering effect that was a function of time, temperature, and heat generation rate.

The appearance of the UO_2 was not the same in all the elements; this is discussed in detail in subsequent sections. Initially, it appeared that the UO_2 sintered into a rather dense layer that was not always in contact with the cladding or MgO but alternately touched both. As burnup proceeded and fission gases were produced, they began to agglomerate and produce pores within the UO_2 layer. The UO_2 expanded until it filled the 0.010-in. annulus but did not exert sufficient force upon the stainless steel cladding to cause it to

deform, nor was the low-density MgO core damaged by the expansion. When the UO_2 came in contact with the stainless steel, its operating temperature was lowered by better heat removal. Even at very high burnups, the steel was able to restrain the further swelling of the UO_2 caused by gas release to the internal pores. Figures 6, 7, 8, 9, and 10 show the UO_2 in elements with burnups of 4 to 52% U^{235} fission. The burnup at which the UO_2 has become sufficiently porous to expand into contact with the cladding to a major degree is dependent upon the initial helium atmosphere (sometimes found to be incomplete because of air contamination during end-plug attachment), the surface temperature of the element, the heat generation rate, and the time at temperature.

The UO_2 particles in the dispersion type elements, as fabricated, ranged from a highly fractured to a nearly unfractured state, depending on the method of fabrication. After irradiation, the UO_2 particles in these elements also experienced marked sintering. The point at which sintering is complete, such that no evidence of previous fractures remain in the UO_2 particles, depends on other factors besides irradiation time, element surface temperature, and heat generation rate. Such factors as the type of matrix material, the particle size of the UO_2 , and the porosity in and around the UO_2 particles are all known to have effect and there are probably other variables such as the oxidation state of the UO_2 and the additives (put in the UO_2 for increased strength) that also have an effect. Figure 11, showing unirradiated UO_2 , can be compared to Figures 12 and 13 which show typical sintering that occurs during irradiation. The effect of these variables on the observed microstructure of the dispersion type elements is described in subsequent sections.

Porosity of UO_2

The UO_2 can exhibit a high degree of porosity at moderate to high burnups as shown in Figures 11, 12 and 13. In every specimen that was metallographically examined, there was evidence of this porosity, although it was difficult to detect at low burnup and low temperatures. Often there were two types of pores, residual amounts of the original irregular pores and the spherical pores created by fission-gas agglomeration. Under certain conditions the pores were nearly invisible at magnification as great as 1000X.

As the pores continue to collect fission gases, the UO_2 expands significantly if not confined. If the UO_2 is tightly held, its operation is relatively cool compared to the unconfined case and the buildup of gases within these pores is reduced. At high burnups and long irradiation times the gas pressures become sufficiently great that the UO_2 expands with sufficient force to produce swelling of the element. A high-strength matrix delays this but at some high burnup or long period of time, swelling could be predicted.

See Figure 18 for an example of swelling of an overheated element. The stability of this type of element is influenced by the choice of matrix as discussed under Matrix Material. As more fission gases collect in the UO_2 pores, they increase in size as well as become spherical. The pores also appear to grow by cracking through the UO_2 that separates the pores. Figure 14 shows a case where this apparently occurred and where it is expected to occur. Except for this type of effect, which produces figure-8 shaped voids, the UO_2 particles are nearly spherical. The combined effects of internal gas pressure, surface tension of the UO_2 , and diffusion combine to produce this spherical shape regardless of the initial shape of the void.

In the case of the annular element, it is usually observed that there is a gradient in pore size across the 0.010-in. layer of UO_2 . This apparently results from differences in temperature across the layer, which may be several hundred degrees at high heat-generation rates. A typical example is shown in Figures 9 and 10 where the gradient is readily detectable. Differences in temperature occurred around the circumference of a fuel element when more than one element was irradiated in a slug. When the elements so irradiated were examined, it was found that the UO_2 porosity varied considerably across the diameter of the same element. The particles of UO_2 in the dispersion elements do not have a large temperature gradient across their diameters and no porosity effect was observed that could definitely be attributed to temperature gradients. There was sufficient variability in the UO_2 particles used in these dispersions that no temperature effects across the element diameter could be observed. It was possible to observe two adjacent UO_2 particles of the same size and yet with different degrees of porosity. See Figure 18. This appeared as a difference in pore size and distribution. If all the UO_2 particles were exactly alike, an effect of temperature would be expected since the ΔT across these elements is approximately 180°C at maximum design heat-generation rates.

One of the irradiated dispersion elements that was examined had UO_2 which contained 0.5 wt % TiO_2 and its behavior was considerably different from the UO_2 with no additives. There were marked differences in porosity within individual particles; some areas exhibited prominent porosity and other areas showed no apparent porosity. Figure 15 shows how the UO_2 varies in appearance even within a single particle. It is not known why this occurs but it may be an effect of TiO_2 on the extent of the UO_2 defect structure and/or UO_2 density. The TiO_2 acts much like a binder between UO_2 particles, as shown in Figure 16, and an unknown amount may enter the UO_2 lattice. It is known that if UO_2 containing TiO_2 is fired too long, at too high a temperature, the over-all density of the particle is lessened by vaporization of TiO_2 and resultant bloating of the sample.² It is believed that this sample operated at high enough temperatures and for sufficient time for bloating to occur in these UO_2 particles in

the zones of high TiO_2 . This is supported by evidence drawn from Figure 17 which shows a network of porosity throughout a particle. This effect is not observed in a majority of the particles however, and the average particle appears more like that shown in Figure 15. The amount of TiO_2 that may have entered the UO_2 lattice and the maximum local density reached in any UO_2 particle are not known; either could be responsible for the variations in the porosity.

One of the dispersion elements examined had operated at a much higher surface temperature than the 600°C that was desired. A precise figure for this temperature is not available but the estimate is in excess of 1000°C . The burnup was 12%* at the point where the metallographic specimen was taken and the element had increased in cross-sectioned area by >20% because of gross UO_2 swelling. The element ruptured and one wide crack extended to the center of the element. An area adjacent to the fractured surface is shown in Figure 18. Because of this fracture, several interesting observations could be made on the behavior of UO_2 during irradiation.

1. The UO_2 particles were highly porous, more so than any other UO_2 from dispersion elements that were examined. The amount of porosity compares with that of the UO_2 in the annular elements which were initially 60% of theoretical density. Since the core of the element contained 20 wt % UO_2 , the particles of UO_2 must have expanded an average of 125% in volume to produce the observed swelling and the resultant UO_2 particles with bulk densities less than 50% of theoretical.
2. There was a marked difference in porosity between the UO_2 particles, as can be seen in Figure 18. This element split so badly that it caused some UO_2 particles to be exposed to the Nak coolant and to be essentially unconfined while other particles were restrained by the stainless steel matrix.

Figures 19 and 20 show UO_2 particles that were unconfined (subsequent to element rupture) and continuously confined, respectively. It is immediately obvious that the pore size is very much different as is the bulk density of the UO_2 . The UO_2 particle of Figure 19, after failure of the element, probably operated at about the same temperature as that of Figure 20, yet the difference in type and amount of porosity exists. This demonstrates the gross swelling of UO_2 that can be expected if there is no restraining force as well as how a relatively small confining force makes a great difference in the swelling of the UO_2 .

*Burnup = % U^{235} fissioned.

3. The UO_2 particles in this cross section exhibited plastic flow during irradiation. The high temperatures, coupled with the high fission density, apparently allowed the UO_2 to flow rather than fracture as would be expected of a brittle material. Examples of the flow are shown in Figures 21 and 22, where UO_2 particles adjacent to the fractured surface are flowing through a narrow opening in the metal matrix into the NaK coolant. The lenticular shape of the pores as well as the abrupt change in pore size at the point of restriction indicate that flow is definitely taking place. The same effect was observed in a dispersion tensile specimen which was irradiated at $\sim 200^\circ\text{C}$ and to 21% burnup. This is demonstrated in Figure 23 where the porosity of the UO_2 increases with proximity to the surface of the element. The restraining force on particles exposed to the coolant is the strength of the UO_2 itself and at this low temperature it swells much like the UO_2 shown in Figure 19.

Growth of UO_2

As mentioned in the previous section, UO_2 may exhibit gross swelling if irradiated at high temperatures and unconfined. This decrease in bulk density is largely due to the collection of fission gases in pores, which causes the UO_2 to expand. Since the magnitude of this growth is so dependent upon the temperature of the UO_2 , the burnup of the UO_2 , and the irradiation time, it was desirable to determine the growth of UO_2 resulting from fission solids only. Therefore, the UO_2 growth in an annular element was investigated in the following manner:

The element, as fabricated, had UO_2 of 63% of theoretical density. After irradiation the cladding had not changed dimensions and, it was subsequently indicated, the MgO center plug was undamaged. A specimen of the element with $\sim 52\%$ U^{235} fissioned was taken for metallographic examination. A typical area of UO_2 was then selected that showed the pores clearly. See Figure 10. A grid was drawn across the sample and the pore fraction of the UO_2 layer was determined by linear analysis. An average of all the resulting values indicated that 32% porosity remained in the UO_2 at this burnup. This is so close to the original porosity of the UO_2 that an accurate growth rate cannot be determined but it does indicate a much smaller value than theoretically predicted. A value of 25% porosity is considered a conservative value. The UO_2 would then have a growth rate of $< 1/3\%$ expansion per cent of uranium atoms fissioned.

The growth rate was calculated for the UO_2 of an element that released an average (over 8-1/4 in. fuel length) of 30% of the fission gases generated. Fission gases were also liberated from the UO_2 lattice to the pores but the extent of this has not been measured. A higher growth rate would be expected for UO_2 that retains the fission gases within its lattice to the highest possible degree.

Similar data cannot be obtained from the dispersion elements because of the wide range of UO_2 particle size and density. The UO_2 particles cannot be measured before and after irradiation, but if the element swells considerably, a rough value of growth can be obtained. This is based on the assumption, of course, that there is no void space in the system to accommodate UO_2 growth without external swelling. This is not the case in any of the dispersion elements irradiated at KAPL since the element densities ranged from 90 to 98% of theoretical.

Porosity is most frequently present in and around the UO_2 particles but the elements fabricated by the cold-binder technique also have measurable porosity within the stainless steel matrix. Figure 24 shows how this porosity is predominantly within the UO_2 or within the matrix metal rather than at the UO_2 - steel interface. Elements fabricated by drawing or rolling generally have the bulk of the porosity at the UO_2 - steel interface. This is considered desirable because the UO_2 will swell to fit the space with a resultant lowering of the UO_2 density and space for gas containment at lower pressures.

The existence of porosity within the steel matrix does not guarantee that swelling will be delayed by the reduction of internal porosity. Some elements exhibited external growth when considerable amounts of internal porosity remained. An example of this is shown in Figures 24 and 25 which are pre- and postirradiation photomicrographs of identical elements. Note the large amount of porosity remaining in the matrix after the element had swelled to 1 to 2 mils in diameter. It was generally observed that the porosity within the matrix was eliminated before any changes in dimension were observed. The conditions under which swelling is delayed by the reduction of internal porosity are not known but irradiation time and temperature are probably important factors. In contrast to Figure 25, Figure 26 shows no external swelling because of the elimination of internal porosity.

The dispersion elements discussed above contained coarse UO_2 particles, 100 to 200 μ in diameter. A considerably different situation existed in an element that contained UO_2 of fine particle size because of the high percentage of fission fragments that recoiled into the steel matrix from the UO_2 particles. The growth rate of UO_2 resulting from fission product solids is quite small and if a large percentage of the fission products escapes, the UO_2 particles tend to shrink. The temperature of the UO_2 then increases and it swells until it is restrained by the matrix, which results in a decrease in the bulk density of the UO_2 . UO_2 sinters and becomes porous much sooner than coarse-particle dispersion elements. Figure 27 shows an element irradiated to 3% burnup that shows this porosity. Additional evidence for the shrinkage of fine UO_2 particles is discussed under Matrix Material.

UO₂ Cracking

The thermal conductivity of irradiated UO₂ is not known but is almost certainly greater than that of the fission gases krypton and xenon. If the UO₂ fractures during irradiation, the resistance to heat flow across the boundaries will be large, even if the fission gases fill the gap. If the cracks are parallel to the direction of heat flow there will be no consequences but, if not, there may be large temperature drops across these gaps. Such cracks (perpendicular to the direction of heat flow) have been observed in some of the annular oxide elements that were metallographically examined. The cause of the cracking is not entirely thermal stresses since such cracks would be radial in nature and this was not observed. It is possible that the differential expansion between the UO₂ (after sintering from loose powders to a solid mass) and the stainless steel tubing is the cause of the cracking. In support of this, the UO₂ layer in the annular elements were often observed in contact with the cladding on one side of an element and separated from the cladding on the opposite side. Figure 28 shows an element in which this occurred.

The UO₂ that was not in contact with the cladding operated at much higher temperature and became much more porous than the UO₂ that was in contact with the cladding. A crack sometimes formed when UO₂ "adhered" to the cladding and the UO₂ pulled away from the cladding upon cooling. This is shown in Figure 29. The sample was irradiated to 4% burnup and no porosity was detectable in the UO₂ other than the irregular included pores. At higher burnups, where pores became pronounced, the cracks tended to close up, as shown in Figure 30. This crack extended to one-third of the circumference of the element. Note also, the difference in the pore size across the crack, which indicated an appreciable temperature drop. The same effect on pore size can be produced by a large void in the UO₂ such as that shown in Figure 31.

Another method of producing cracking of the UO₂ might be high fission-gas pressures in the pores. This method would result in more random cracking than that resulting from thermal stresses and would propagate by fracturing the UO₂ membranes between the pores. This type of crack is shown in Figure 10.

The same area at high magnification, shown in Figure 32, indicates that the fractured surface smooths itself during irradiation. Post-irradiation cracking is easily distinguished from irradiation cracking because of this effect. Figure 33 shows both types of cracks. The UO₂ layer in this type of element is not fractured as badly as might be expected; this is probably because of the ability of the normally brittle UO₂ to deform, rather than fracture, when under irradiation. Why it will swell in some cases and crack in others is not thoroughly understood.

No cracking of UO_2 particles that could be attributed to thermal stresses has ever been observed in the dispersion elements. Power cycling produces no measurable effect regardless of temperature or heat generation rates.

Matrix Material

The stability of an element containing UO_2 as the dispersed fuel is dependent on the properties of the matrix material. Dispersions fabricated from pre-alloyed Type 347 stainless steel powders as well as 18Cr-12Ni-70Fe and 16Cr-84Fe powders were irradiated. Some unusual effects that resulted from the use of the pre-alloyed Type 347 stainless steel powders are described in this section, on pages 25 and 26.

Important differences exist between the physical properties of austenitic 18-12 steel and 16Cr ferritic steel.* The coefficient of thermal expansion of the austenitic steel is considerably greater than that of UO_2 . Assuming that the UO_2 particles are in perfect contact with an unstressed austenitic steel matrix, they will not remain so as the sample comes to temperature. The thermal resistance of a UO_2 -steel interface can be appreciable and as the matrix tends to expand away from the UO_2 , the UO_2 must become hotter than the steel. Ferritic stainless steels, on the other hand, have coefficients of expansion that are very near those of UO_2 . There is no tendency for a gap to form and the UO_2 operates at a lower temperature than in an austenitic matrix. Also, the matrix temperature is lower in a ferritic steel element because of higher thermal conductivity.

The conditions stated above apply if the matrix is unstressed and in contact with the UO_2 at room temperature. The validity of such an assumption depends on the method of fabrication. In the case of the cold binder elements, which enter and leave the swaging dies at 1000 and 600°C, respectively, this assumption is reasonable. At ~600°C the UO_2 and the steel should be in contact and as the sample is cooled to room temperature the steel shrinks against the UO_2 . For 0.008-in. UO_2 particles the strain put into the steel is:

$$(\alpha_{\text{steel}} - \alpha_{\text{UO}_2}) \Delta T = (19-10) \times 10^{-6} (600) = 0.0054 = 0.54\%$$

This is greater than the yield (assumed to be 0.02%), therefore the net strain is considered as 0.02%. This is relieved almost immediately as the sample is heated and continued heating results in the formation of a gap between the UO_2 and the steel. As the maximum core temperature for this element is approximately 800°C, a separation of approximately 0.03 mil is predicted. The fission

*Both steels were fabricated with carbonyl iron and nickel powders and electrolytic chromium powders. The total impurity content was negligible and the final carbon content was determined as less than 0.01%.

gases krypton and xenon exist at this interface and the temperature drop is only slightly dependent on the percentage released because the conductivity of these gases varies little with temperature and pressure.³ It is estimated that a UO_2 particle 200 μ in diameter in an austenitic steel fuel element with 20% burnup need release only 0.01% of its fission gases to produce one atmosphere at the interface. This is sufficient to cool the UO_2 so that the temperature drop across the interface is $\sim 50^\circ\text{C}$. This assumes no heat removal by radiation (which is approximately true) and no heat removal by conduction from the UO_2 directly to the steel. The final conclusion is that the UO_2 operates at a temperature somewhat greater than that of the surrounding matrix in an austenitic steel dispersion. In the ferritic steel dispersion the matrix is at a lower temperature than the austenitic element and the UO_2 is at essentially the same temperature as the matrix.

In both the annular and the dispersion types of elements, stainless steel is immediately in contact with UO_2 . The fission fragments have sufficient energy for a layer of stainless steel in contact with the UO_2 to become highly damaged as a result of the effects of the fission spikes and the resultant impurity atoms. In the unetched condition no evidence of a damaged zone in the steel has ever been detected but after etching such a zone is always present. Figures 34 and 35 show typical damaged zones in the two types of elements. The damaged layer is sufficiently different from the remaining stainless steel of the matrix that it does not exhibit a normal etched structure. In many cases a slip-line grain boundary can be followed up to the damaged zone where it disappears as shown in Figure 35. A similar dispersion element with a high burnup has shown a slightly different effect. The damaged zone is visible but when a grain boundary is followed from the matrix into this zone the boundary widens considerably. It appears as if the steel is tearing apart at the original grain boundaries. This effect is seen only on high burnup samples and is probably due to UO_2 growth with burnup. Figure 36 shows a sample with 30% burnup that demonstrates this effect.

In examination of the broken end of an unclad tensile specimen it was apparent that the damaged zone is brittle. Figure 37 shows matrix metal that has been substantially elongated during the tensile testing and evidence of cold-work can be seen. The sample was pulled at room temperature and the small amount of slip planes visible agrees well with room temperature tests on unirradiated dispersions. The two large voids on either side of the strip are areas where UO_2 particles originally resided. In testing, the UO_2 was crushed and during polishing it dropped out. It is evident in Figure 37 that the damaged layer of steel is discontinuous and apparently quite brittle.

In a joint KAPL - BMI program, dispersion type fuel was annealed to determine the effect of time (at various temperatures) upon density, dimensions, and fission-gas release.⁴ The specimens were annealed at temperatures as high as 900°C for 200 hr. Metallographic examination of two elements revealed no detectable change in the low-density hot-drawn element, but significant change in the high-density hot-swaged element. The net decrease (3-1/2%) in density of the cold-binder element was sufficiently large for an effect of annealing to be proven by metallographic examination. Figures 38 and 39 show this element as irradiated and as annealed, respectively. Note that void space has been created at the UO₂ - stainless steel interface and that the UO₂ particles are not tightly held after annealing. Numerous cracks were produced in the steel matrix around the UO₂ particles as shown in Figure 40. The annealing produced sufficient gas pressure to cause permanent deformation of the steel as well as cracking. This cracking is quite different from that shown in Figure 18 and of different origin: gas pressure from released fission gases rather than UO₂ growth.

The UO₂ of the annealed specimen is definitely more porous than the irradiated specimen. The two elements were 1-1/2-in. long, were cut from the same element for irradiation, and were irradiated side by side in the same capsule. Hence, they were as nearly alike as possible before annealing. If it is assumed that they are identical after irradiation, the UO₂ (as well as the matrix) has definitely been affected by the 200 hr anneal at 900°C.

The change in porosity in the UO₂ is the result of fission-gas collection in the pores, which indicates that the UO₂ operated below 900°C during irradiation. It is likely that the pores would have increased much more in size if the UO₂ was under irradiation, at this high temperature.

The chemical properties of the damaged layer of steel differ from those of the normal stainless steel. The etchant attacks this layer at a faster rate than elsewhere and continued etching results in complete removal of the damaged zone. Typical etching bands in a cold binder element are shown in Figures 34 and 36. A considerably different effect is seen in slightly different elements. Figures 41 and 42 are photomicrographs of an element that contains fine-particle-size UO₂ irradiated to 21% burnup. The element had 91% of theoretical density, and, because of the process used, the original UO₂ of 44 to 74μ was reduced to approximately 20 to 40μ. This element was fabricated of pre-alloyed Type 347 stainless steel powders while the majority of the elements irradiated at KAPL were made of 18Cr-12Ni-70Fe powders of high purity.

These elements have approximately 1% silicon in the Type 347 stainless steel and the presence of the silicon produced a rather unique result. During irradiation the large majority of the fission fragments recoiled from the UO_2 into the steel. This resulted in sufficient loss of atoms that the UO_2 had a tendency to shrink in size but this is counteracted by the tendency to expand until restrained. Since most of the fission fragments recoil into the steel there is an abundance of oxygen released by U^{235} fission. If the oxygen were not used up somehow, the UO_2 would go to a higher oxidation state, provided the temperature was not so high as to cause instability of higher uranium oxides. The oxygen cannot go into solution in the steel because an adherent film of Cr_2O_3 protects the underlying metal, and internal oxidation of the steel is prevented.

The most stable oxides of the elements present in the steel are SiO_2 , MnO , and Cr_2O_3 . The Cr_2O_3 normally forms a protective layer that effectively stops further oxidation but the silicon reduces Cr_2O_3 and forms a subscale that breaks up the protective coating. As the silicon is depleted at the surface, a concentration gradient is formed and silicon diffuses into the depleted areas, where it is oxidized. At higher burnups, the UO_2 continues to lose atoms by recoil and the oxides continue to build up until, eventually, the UO_2 particles are embedded in oxides rather than steel. This should increase the operating temperature of the UO_2 beyond that of a coarse-particle dispersion but an accurate temperature value cannot be calculated.

The free energy of formation of SiO_2 is too great for the UO_2 to reduce it by going to a higher state of oxidation; hence, only the dioxide of uranium should be present. The logical question that arises is what happens to the fission fragments that recoil into the steel. Many of these atoms such as the rare earths form more stable oxides than SiO_2 , and would they not diffuse and become oxidized in a like manner? An investigation was conducted by etching the samples and collecting the drippings for analysis. This was possible because this foreign phase is rapidly removed by the aqua regia etchant. Upon analysis, the solution was found to contain Fe and Si in the ratio of 3 to 1 whereas the original steel has a ratio of 88 to 1, which demonstrates that silicon is removed selectively from the sample. This would not be the case during a stainless steel etch, thus verifying that silicon is a major component of the dissolved phase. Gamma scanning of the solution produced prominent peaks at Cs^{137} and Ce^{144} but both were present in very small quantities compared to silicon. Cs^{137} is 0.1% of the silicon content and Ce^{144} much less. This indicates that fission fragments have not concentrated in the second phase and agrees with the observations on all other elements that contained no silicon and where no second phase had been detected.

Two different samples, before and after etching, are shown in Figures 41 to 44. In both cases the second phase was removed by etching to leave spherical UO_2 particles that were loosely held by the matrix. The two samples received 21 and 44% burnup and operated at 600 and 300°C element surface temperatures, respectively. The amount of UO_2 that remained in the sample shown in Figure 43 was small in comparison to the quantity of the other oxides present. All samples of this type exhibited the same phenomenon while none of the coarse-particles samples exhibited such an effect. The large UO_2 particles are actually deficient in oxygen whereas the small UO_2 particles have an excess. The lack of oxides at the UO_2 - steel interface in the coarse-particle dispersion could occur because no free oxygen is released from the UO_2 particles. The oxidation of fission products could then occur only by solid-state reaction between the UO_2 particles and the surface oxides of the stainless steel.

Defected Elements

Several irradiations were performed on defected elements of the annular and dispersion type to determine how severe an accessibility problem would result from various types of failures. A typical defect of the annular oxide element was a hole drilled through the cladding or a leak resulting from a faulty end-plug.⁵ The size of defect ranged from a hole so small as to be detectable only by a mass-spectrometer leak detector up to a 0.009-in. drilled hole. These elements were cycled into and out of pile to duplicate reactor pile power changes. It was expected that NaK would be drawn inside the element by the cycling and remove fission fragments or cause an element failure. Out-of-pile tests indicated that power cycling of elements with 0.003 to 0.009-in. holes drilled through the cladding resulted in rupture and longitudinal splitting of the cladding. The cracks did not originate at the holes but were always in that vicinity.⁶ Some of the elements became completely filled with NaK even though the holes were drilled at the center of the element and the elements were cycled in a vertical position. The damage was so severe that some elements were broken in two.

In-pile cycling, however, showed that no such effect takes place. The defected elements did not fill with NaK (used in place of sodium in all irradiation assemblies) as shown by weight and center-of-gravity measurements, cross-sectioning and metallographic examination of the elements. Metallographic examination of typical annular elements indicated that the out-of-pile cycling tests were meaningless because soon after insertion the UO_2 sintered from loose-packed powders to a solid mass. The sintered UO_2 apparently does not absorb NaK in the same manner as the loose-packed powders. Figure 45 shows how a slight amount of NaK penetrated an element that received 9% burnup and >1000 power cycles. The penetration of NaK through the drilled hole defect

could not be detected by weight gain, center of gravity, sectioning, and immersion in water containing phenolphthalein, or visual examination of a cross section. The photomicrographs revealed that a slight amount of NaK had entered the hole but did not travel more than $1/4$ in. to either side of the defect. The thin dark layer adjacent to the cladding in Figure 45 is considered a compound of sodium and uranium oxides but its composition has not been determined. In any event the amount of NaK that entered the element was very small as a result of the sintering of the UO_2 . Hence, the sintering must take place almost immediately since the power cycling would tend to draw NaK into the elements at the beginning of cycling. This is supported by observations of other annular elements which showed that the UO_2 sintered before 4% burnup. See Figure 6.

Other annular oxide elements with a variety of types of defects were irradiated and the results indicated that the elements should contain their fission products much better than originally anticipated. A portion of the length of some of the elements melted because of lack of coolant and the unmelted portion of the element retained its fission products quite well.⁵

Irradiations were also performed on dispersion elements with various types of defects. This type of element was expected to create a minimum accessibility problem in the event of failure. The defected dispersion elements were generally irradiated without end plugs or with slots milled through the cladding. This type of element was also shown to be excellent in retaining fission products when the core is exposed to NaK.

Two β capsules that contained dispersion elements developed leaks and lost part of the NaK coolant. One element melted down about one inch out of the three and metallographic examination was performed adjacent to the melted end. Except for heavy sensitization of the Type 304 stainless steel cladding, the cross section did not appear significantly different from others. Figure 46, showing a section cut $1/8$ in. from the melted end, demonstrates this. The UO_2 was apparently not affected by the high temperatures, probably because the temperature was achieved by lack of cooling and not by a high fission density in the UO_2 . The high fission density is considered as important in causing the UO_2 to sinter and become porous as the effects of temperature. The length of the element projecting above the coolant is not known nor is the operating temperature at the observed cross section. It was, however, quite hot, as demonstrated by the cladding sensitization and the proximity to the melted end.

Additional evidence to support the statement that fission density is as important as temperature in producing sintering and porosity in UO_2 can be obtained from examination of dispersion tensile test specimens. These specimens were unclad UO_2 - steel dispersions, 0.100-in. in diameter and were irradiated at surface temperatures of $\sim 200^\circ C$. The UO_2 sintered and became

porous at high burnups in spite of the low irradiation temperatures, as shown in Figure 47. The UO_2 was only lightly fractured by the tensile testing because it was $1/8$ in. from the broken end. The UO_2 particles at the ruptured end were highly fractured as shown in Figure 48 but the UO_2 did not fall out in appreciable quantities during polishing because the UO_2 swelled against the stainless steel matrix during irradiation. The UO_2 particles were in intimate contact with the steel even after tensile testing, as if there were a bond between the two. Figure 49 shows this effect.

CONCLUSIONS

Many irradiation effects in UO_2 were observed and a theory about the behavior of UO_2 was established. The role of fission gases in UO_2 performance is apparently very important and additional work on diffusion coefficients and activation energies should be performed. Additional knowledge of the size and location of the fission fragments as they exist within the UO_2 lattice will be necessary before the radiation behavior of UO_2 is thoroughly understood. Such a knowledge might enable us to extend the capabilities of UO_2 beyond that already demonstrated or to develop other systems of even better irradiation stability.

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ILLUSTRATIONS

NOTE: All photographs have been reduced 50% from stated magnification for facility of publication.

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FIGURE 1 FLOOR LAYOUT OF REMOTE METALLOGRAPHY CELL

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FIGURE 2. FRONT FACE OF THE METALLOGRAPHIC CELL SHOWING THE THREE MODULES, IN WHICH THE PROCESSING OF THE SAMPLES IS ACCOMPLISHED, AND THE MANIPULATORS USED FOR REMOTE OPERATION. TWO OF THESE MANIPULATORS ARE USED FOR EACH MODULE. THE CONTROL PANELS LOCATED ON THE CELL WALL ARE FOR REMOTE OPERATION OF THE EQUIPMENT IN THE CELL.

FIGURE 3. TWO BUCHLER POLISHERS WITH AUTOMET ATTACHMENTS, COOLANT DISPENSERS, 5-HOLE SPECIMEN HOLDER, SPECIMEN LOADING ASSEMBLY, PRECISION-JARRETT PRESS, AND AN ALSOP FILTERING UNIT. THESE POLISHERS MAY BE USED FOR EITHER GRINDING OR POLISHING. THE ALSOP FILTERING UNIT IS USED TO FILTER OUT THE RADIOACTIVE MATERIAL FORMED DURING THE GRINDING AND POLISHING OPERATIONS.

FIGURE 4. THE EQUIPMENT USED FOR ETCHING AND CLEANING THE METALLOGRAPHIC SAMPLES. INCLUDED ARE THE HOT AIR HAIR DRYER, VACUUM HOOD, 5-HOLE SPECIMEN HOLDER AND SPECIMEN LOADING ASSEMBLY.

FIGURE 5. SHIELDED BAUSCH AND LOMB RESEARCH METALLOGRAPH USED TO EXAMINE AND PHOTOMICROGRAPH THE IRRADIATED SPECIMENS. THE CONTROLS TO OPERATE THE VARIOUS MOVEMENTS OF THE METALLOGRAPH ARE LOCATED ON THE FRONT OF THE SHIELDING. THE LEAD GLASS WINDOW WAS INSTALLED FOR VIEWING THE STAGE. THE DOOR AT THE REAR OF THE SHIELDING MAY BE OPENED TO ALLOW THE CASTLE MANIPULATOR TO BE EXTENDED INTO THE CELL INTERIOR TO PICK UP THE SPECIMEN AND PLACE IT ON THE STAGE OF THE METALLOGRAPH. THE LEAD DOOR, IN THE FRONT SHIELDING, MAY BE OPENED FOR ACCESS TO THE STAGE OF THE METALLOGRAPH.

FIGURE 6. UO_2 IRRADIATED TO 4% BURNUP. 500X, AS ETCHED.

FIGURE 7. UO_2 IRRADIATED TO 9% BURNUP. 100X, AS POLISHED.

FIGURE 8. UO_2 IRRADIATED TO 25% BURNUP. 500X, AS POLISHED.

FIGURE 9. UO_2 IRRADIATED TO 36% BURNUP. 250X, AS POLISHED.

FIGURE 10. UO_2 IRRADIATED TO 52% BURNUP. 100X, AS POLISHED.

FIGURE 11. UNIRRADIATED UO_2 PARTICLE OF $\sim 95\%$ DENSITY IN STAINLESS STEEL MATRIX. 500X, AS ETCHED.

FIGURE 12. UO_2 IRRADIATED TO 12% BURNUP. 500X, AS ETCHED.

FIGURE 13. UO_2 IRRADIATED TO 30% BURNUP. 500X, AS POLISHED.

FIGURE 14. UO_2 IRRADIATED TO 52% BURNUP. 300X, AS POLISHED.

FIGURE 15. UO_2 IRRADIATED TO 16% BURNUP AND ANNEALED AT 900°C FOR 200 HOURS. 250X, AS POLISHED.

FIGURE 16. SINTERED UO_2 PARTICLE CONTAINING TiO_2 AS A STRENGTHENER.

FIGURE 17. UO_2 IRRADIATED TO 16% BURNUP AND ANNEALED AT 1600°F FOR 200 HOURS. 500X, AS ETCHED.

FIGURE 18. DISPERSION ELEMENT IRRADIATED TO 12% BURNUP AT $\sim 1000^{\circ}\text{C}$. 50X, AS ETCHED.

FIGURE 19. UO_2 PARTICLE IRRADIATED TO 9% BURNUP. UNCONFINED SUBSEQUENT TO ELEMENT RUPTURE. 250X, AS ETCHED.

FIGURE 20. UO_2 PARTICLE IRRADIATED TO 9% BURNUP. CONTINUOUSLY CONFINED. 200X, AS POLISHED.

FIGURE 21. PLASTIC FLOW OF UO_2 TOWARD FRACTURED SURFACE. 150X, AS POLISHED.

FIGURE 22. PLASTIC FLOW OF UO_2 TOWARD FRACTURED SURFACE. 250X, AS ETCHED.

FIGURE 23. PLASTIC FLOW OF UO_2 IRRADIATED TO 21% BURNUP AT $\sim 200^{\circ}\text{C}$. 100X, AS POLISHED.

FIGURE 24. UNIRRADIATED UO_2 PARTICLE DISPERSED IN STAINLESS STEEL. 100X, AS ETCHED.

FIGURE 25. UO_2 PARTICLE IRRADIATED TO 30% BURNUP. 100X, AS POLISHED.

FIGURE 26. UO_2 IRRADIATED TO 25% BURNUP. 100X, AS POLISHED.

FIGURE 27. UO_2 IRRADIATED TO 3% BURNUP. 500X, AS ETCHED.

FIGURE 28. UO_2 IRRADIATED TO 9% BURNUP. 100X, AS POLISHED.

FIGURE 29. UO_2 IRRADIATED TO 4% BURNUP. 100X, AS POLISHED.

FIGURE 30. UO_2 IRRADIATED TO 36% BURNUP. 200X, AS POLISHED.

FIGURE 31. UO_2 IRRADIATED TO 24% BURNUP. 200X, AS POLISHED.

FIGURE 32. UO_2 IRRADIATED TO 52% BURNUP. 300X, AS POLISHED.

FIGURE 33. UO_2 IRRADIATED TO 36% BURNUP. 500X, AS POLISHED.

FIGURE 34. DAMAGED ZONE AROUND UO_2 PARTICLES IRRADIATED TO 25% BURNUP. 20X, AS ETCHED.

FIGURE 35. STRUCTURE OF DAMAGED STEEL LAYER. 500X, AS ETCHED.

FIGURE 36. TEARING AT ORIGINAL GRAIN BOUNDARIES OF DAMAGED STAINLESS STEEL LAYER. 1000X, AS ETCHED.

FIGURE 37. DEMONSTRATION OF BRITTLINESS OF DAMAGED STEEL LAYER. 500X

FIGURE 38. UO_2 AS IRRADIATED TO 8% BURNUP. 500X, AS POLISHED.

FIGURE 39. UO_2 IRRADIATED TO 9% BURNUP AND ANNEALED 200 HOURS AT 900°C . 500X, AS POLISHED.

FIGURE 40. TEARING OF MATRIX DUE TO HIGH FISSION GAS PRESSURES DURING 900°C ANNEAL. 100X, AS POLISHED.

FIGURE 41. SECOND PHASE PRODUCED AT UO_2 -STEEL INTERFACE. 500X, AS POLISHED.

FIGURE 42. REMOVAL OF 2ND PHASE BY ETCHING. 500X

FIGURE 43. SECOND PHASE PRODUCED AT UO_2 -STEEL INTERFACE. 250X, AS POLISHED.

FIGURE 44. REMOVAL OF 2ND PHASE BY ETCHENT PARTIALLY COMPLETE. 1000X

FIGURE 45. REACTION BETWEEN NaK AND UO_2 IN A DEFECTED ELEMENT. 500X, AS POLISHED.

FIGURE 46. CROSS SECTION OF A DISPERSION ELEMENT TAKEN $\sim 1/8$ IN. FROM THE MELTED END. 50X, AS POLISHED.

FIGURE 47. UO_2 IRRADIATED AT $\sim 200^\circ\text{C}$ TO 21% BURNUP. 100X

FIGURE 48. UO_2 FRACTURING CAUSED BY TENSILE TESTING. 100X, AS POLISHED.

FIGURE 49. UO_2 AFTER TENSILE TESTING. 500X, AS POLISHED.