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# OFF-DESIGN TEMPERATURE EFFECTS ON NUCLEAR FUEL PINS FOR AN ADVANCED SPACE-POWER-REACTOR CONCEPT

by Kenneth J. Bowles Lewis Research Center Cleveland, Obio 44135

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| formulating a reli   | able reactor s  | afety analysis an       | d designing an eme         | ergency core coo                       | ling system.     |  |
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| showed no compat   | ibility problem   | ns after heating f      | or 8 hours at 2400         | <sup>o</sup> C. At 2520 <sup>o</sup> C | and above,       |  |
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# OFF-DESIGN TEMPERATURE EFFECTS ON NUCLEAR FUEL PINS FOR AN ADVANCED SPACE-POWER-REACTOR CONCEPT by Kenneth J. Bowles

Lewis Research Center

# SUMMARY

An exploratory out-of-reactor investigation has been conducted to determine the behavior of simulated fuel pin materials for an advanced nuclear space-power concept during short-time temperature excursions above the nominal operating temperature of about  $990^{\circ}$  C. Initial compatibility and fuel redistribution information required for the formulation of a reliable reactor safety analysis and for the design of an emergency core cooling system was obtained.

Eight simulated uranium mononitride (UN) fuel pins clad with tungsten-lined T-111 (Ta-8W-2Hf) were heated in vacuum to temperatures ranging from  $1800^{\circ}$  to  $2700^{\circ}$  C for times varying from 1 minute to 8 hours. Metallography, scanning electron microscopy, cladding ductility tests, and chemical analyses were used in evaluating the test pins.

Fuel pin cladding damage resulting from fuel decomposition occurred in 1 hour at a temperature of  $2520^{\circ}$  C, and, as temperatures increased above this, the time for reaction decreased. For example, at  $2700^{\circ}$  C a portion of the cladding was destroyed in 1 minute. This was caused by attack of molten free uranium, which was accumulated in the fuel pin as a result of decomposition of the UN fuel and loss of nitrogen by permeation through the cladding. Also, the liquid uranium, because it wets the cladding and flows to other portions of the fuel pin, leads to redistribution of the fuel.

Tests at temperatures of  $2400^{\circ}$  C for 8 hours showed no signs of compatibility problems. Fuel redistribution was negligible, and no free uranium was detected either in the fuel or in the liner or cladding. This suggests that a maximum cladding temperature limitation of about  $2400^{\circ}$  C could be assumed for calculation of emergency core cooling requirements. However, the experiments performed here were exploratory in nature and although they approximate the situation, a more detailed investigation of fuel pin geometry effects and time-temperature effects is required for precise design work.

#### INTRODUCTION

Nuclear reactors for space-power application must be designed to incorporate a high degree of safety and reliability. One space-power system concept investigated at the Lewis Research Center uses a fast-spectrum, lithium-cooled nuclear reactor (ref. 1). The reactor core is made up of cylindrical fuel pins consisting of uranium mononitride (UN) fuel clad with tungsten-lined T-111 (Ta-8W-2Hf alloy). These fuel pins must perform at a nominal cladding temperature of about  $990^{\circ}$  C for at least 50 000 hours. A schematic of the reactor concept fuel pin is shown in figure 1. The UN fuel is incompatible when in direct contact with tantalum alloys at the temperature of operation (~990° C) specified for this reactor (ref. 2). For this reason, a thin tungsten liner is interposed between the fuel and the cladding. T-111 alloy caps seal the helium filled pins. T-111 vibration suppressors and tungsten disks separate the UN fuel from the end caps.

Potential reactor malfunctions that would result in temperature excursions have been analyzed (refs. 3 and 4). The most severe malfunction is a loss of coolant (lithium) accident. In thise case the afterheat generated within the fuel pins can drive fuel temperatures to the melting point (ref. 3). If this occurs, the fuel loading within certain zones of the reactor is sufficient to allow a critical mass of uranium to assemble. Therefore, fuel melting must be avoided by incorporating emergency cooling systems into the reactor design. Malfunctions that lead to increased temperatures well above the operating temperatures but below the melting point can also occur. The degree of dissociation of the UN fuel into nitrogen and uranium will increase as the temperature increases from the nominal operating temperature to the melting point of the fuel. The resulting free uranium and nitrogen could cause fuel redistribution and damage to the fuel pin (ref. 5). If such damage were severe, the reactor would have to be shut down permanently.

This test program was intended as an exploratory investigation of fuel pin behavior above the nominal operating temperature to identify information required for the formulation of a reliable reactor safety analysis. The main objectives were

- To determine if and how the fuel will redistribute itself at temperatures near its melting point (e.g., by vapor transport, slumping, or melting)
- (2) To estimate temperature-time levels below which transient temperature excursions would not cause fuel pin damage or fuel redistribution
- (3) To determine whether the cause of fuel pin damage would be liquid uranium corrosion of the cladding, or embrittlement by nitriding of the cladding, or both.

The approach was to heat simulated fuel pins out of the reactor in a vacuum furnace at temperatures from  $1830^{\circ}$  to  $2700^{\circ}$  C and for times of 1 minute to 8 hours. A maximum excursion time of 8 hours was assumed for a reactor lifetime of 50 000 hours. The

10-minute tests were run because this was the estimated period of time for one excursion. The pins then were examined for fuel and cladding structural changes, fuelcladding reactions, cladding contamination, and cladding ductility changes.

#### EXPERIMENTAL DESCRIPTION

### Materials

The T-111 alloy tubing used to prepare simulated fuel pins (capsules) for this series of tests was seamless with a 0.952-centimeter (0.375-in.) diameter and a 0.084-centimeter (0.033-in.) wall. The inside diameter of the tubing was reamed and honed to a 0.808-centimeter (0.318-in.) diameter. The tubing was in the recrystallized condition with an ASTM standard grain size of No. 6 (0.05 mm). The vendor's chemical analysis is shown in table I.

The protective liner was fabricated from 0.003-centimeter (0.0012-in.) thick tungsten foil in the as-rolled condition. The oxygen and nitrogen contents of the as-received tungsten foil are 24 ppm by weight and 2 ppm by weight, respectively.

The UN fuel used in the tests was depleted in the uranium-235 isotope. The fuel was in the form of cylindrical pellets, which were fabricated by uniaxially pressing and sintering the powder to a density of 94.4-percent of theoretical density. Details of the fabrication procedure are given in reference 6. The average analysis of typical UN pellets used in this study is as follows:

| Element  | Composition,                 |  |  |
|----------|------------------------------|--|--|
|          | wt %                         |  |  |
| Uranium  | <b>94.</b> 44 <u>+</u> 0. 14 |  |  |
| Nitrogen | $5.47 \pm 0.04$              |  |  |
| Oxygen   | $0.0173 \pm 0.0026$          |  |  |
| Carbon   | 0.0335 <u>+</u> 0.0048       |  |  |

Figure 2 shows the microstructure of the fuel as fabricated. No second phases are visible at a magnification of  $\times 500$ .

#### **Test Capsules**

The simulated fuel pins (capsules) prepared for these tests were all basically identical. A sketch of the test capsule is shown in figure 3. Each capsule contained one

cylindrical UN fuel pellet measuring 0.787 centimeter (0.310 in.) in diameter and 0.958 centimeter (0.377 in.) long with a 0.254-centimeter (0.100-in.) diameter axial hole. The cladding was made from a 3.17-centimeter (1.25-in.) long piece of T-111 tubing, 0.952 centimeter (0.375 in.) in diameter with a 0.069-centimeter (0.027-in.) wall thickness. To prevent direct contact between the UN and the T-111, a loose wrapping of 0.0030-centimeter (0.0012-in.) thick tungsten foil was inserted into the tubing. This loose cylinder was 1.91 centimeters (0.75 in.) long. For six of the capsules, one layer of tungsten was used to line the cladding inside diameter. In the remaining two capsules, two layers of tungsten were used.

To prevent the fuel pellet from moving axially within the capsule and touching the T-111 end plugs, a thin-walled piece of 0.64-centimeter (0.25-in.) diameter by 0.318-centimeter (0.125-in.) long tungsten - 26-percent rhenium (W-26Re) alloy tubing was used as a spacer at each end of the pellet. Tungsten spacers were not used because tungsten tubing was not readily available for this series of tests and the W-26Re tubing served the purpose. The end plugs were machined from T-111 rod to fit snugly within the cladding. The top end plug was 0.960 centimeter (0.378 in.) long and contained a 0.152-centimeter (0.060-in.) diameter by 0.762-centimeter (0.300-in.) long blackbody to pyrometer sightings.

he T-111, W-26Re, and the tungsten foil were degreased, ultrasonically cleaned

detergent, rinsed with distilled water and ethyl alcohol, and acid etched before capsule assembly. The acid etch consisted of one part hydrofluoric acid (48 percent by weight), four parts nitric acid (70 percent by weight), one part sulfuric acid (95 percent by weight), and two parts distilled water.

After the capsules were sealed by gas-tungsten-arc welding in a helium atmosphere, they were leak checked with a helium mass spectrometer and X-ray radiographed. Until they were heated, the capsules were stored in glass containers to prevent contamination of the surfaces.

# **Test Apparatus and Procedure**

Figure 4 shows a sketch of the vacuum induction furnace with the test capsule in place within it. The test furnace is a vacuum $(1.33 \times 10^{-3} \text{ N/m}^2 (10^{-7} \text{ torr}))$  induction furnace with a water-cooled quartz tube as containment. Temperature measurements were made with an optical pyrometer which was calibrated with a National Bureau of Standards standard lamp. The estimated accuracy of the temperature readings is about  $\pm 0.5$  percent. The cup with the capsule was positioned on the furnace pedestal so that direct line of sight between the pyrometer and the blackbody hole in the top of the capsule could be made through a glass prism on the top of the furnace.

The procedure for heating the capsule was to hold the capsule at  $1000^{\circ}$  C for 1 hour and then raise the temperature to the desired level ( $1800^{\circ}$  to  $2700^{\circ}$  C) as quickly as possible. To increase the temperature to the test level quickly, the furnace was calibrated with a dummy capsule so that an accurate relationship between the furnace power and the capsule temperature could be established. To heat from  $1000^{\circ}$  C to the test temperature, the power was quickly increased to the level that had been found through the calibration to produce the desired capsule temperature. The time from the initiation of this power increase to the attainment of a constant temperature normally took about 1.5 to 2.0 minutes. Test time started when the desired temperature was attained. Some drifting of temperature occurred, but it did not exceed  $\pm 20^{\circ}$  C during any of the tests.

At the end of the test period, the power to the furnace was shut off, and the test capsule was allowed to cool by radiation. The capsule was removed from the furnace 6 hours after the power was shut off. The capsule was then radiographed and checked for leaks with a helium mass spectrometer. Before the capsule was disassembled, the radiographs were examined and compared with the pretest radiographs to locate areas of potential interest for subsequent examination (e.g., to record the position of any reaction zones between the capsule and fuel and to determine the proper procedure for capsule disassembly to preserve areas of interest for examination).

# **Posttest Evaluation Procedures**

The posttest evaluation of the test capsules consisted of metallographic examination of the fuel, liner, and cladding; chemical analysis (oxygen and nitrogen) of the cladding; cladding ductility measurements; and scanning electron microscopy (SEM) on capsule areas where optical metallography showed that reactions had occurred. These procedures have been developed as standard evaluation procedures at Lewis.

The etchants for the metallographic evaluation of the capsule parts are

- (1) For T-111 30 grams of ammonium fluoride, 50 milliliters of nitric acid, and 20 milliliters of water
- (2) For tungsten 10 grams of potassium hydroxide, 10 grams of potassium ferrocyanide  $(K_2Fe(CN)_6)$ , and 20 milliliters of water
- (3) For UN 60 milliliters of lactic acid  $(HC_3H_5O_3)$ , 24 milliliters of nitric acid, and 2 milliliters of hydrofluoric acid.

Inert gas fusion analysis was used for oxygen and nitrogen determination on cladding samples and nitrogen-doped T-111 samples. The sensitivity of the procedure is  $\pm 0.5$ -percent for oxygen and nitrogen determinations. Ductility tests were performed on 0.64-centimeter (0.25-in.) wide rings of the cladding that were cut from the cladding

using a hacksaw. The procedure for specimen preparation is described in reference 7. The ductility was tested by flattening the ring specimen in a vise in a dry argon-filled plastic bag.

Metallographic samples from selected capsules were examined with a scanning electron microscope (SEM). To do this, the samples were polished and etched (where necessary), and a  $20 \times 10^{-10}$  meter (20 Å) layer of gold was vapor deposited on the surface. Standard SEM procedures were then used to examine the prepared surfaces.

### RESULTS

Eight capsules were tested by simulating overtemperature excursions, which possibly could occur in the core of the reactor concept under study. In addition to these capsule tests, six samples of T-111 tubing doped with nitrogen were exposed to the same test conditions as those capsules in which reactions occurred during testing. The purpose of exposing the doped samples to the test environment was to get an indication of the stability of nitrides (i.e., HfN and TaN) in T-111.

The test conditions and results for the capsule tests are given in table II, and the results of the stability tests on nitrogen-doped T-111 tubing are given in table III. No attempt was made to measure nitrogen partial pressures in the vacuum furnace at the test temperatures nor was any attempt made to determine nitrogen gradients within the cladding test specimens after the tests were completed.

For the short (10 min) tests at temperatures of  $2500^{\circ}$  C and below (capsules 1, 2, 3, and 5), no evidence of fuel, liner, and cladding reaction was visible from photomicrographs or X-ray radiographs. Also, for the 480-minute test at  $2400^{\circ}$  C (capsule 4), there was no evidence of reaction. But some bonding occurred between the fuel and the liner at test temperatures of  $2500^{\circ}$  C and below.

Visual examination of capsules 6  $(2520^{\circ} \text{ C}, 60 \text{ min})$  and 7  $(2600^{\circ} \text{ C}, 10 \text{ min})$  showed little change to the outer surface of the cladding. But some thermal etching, revealing an enlarged grain structure, was evident. Capsule 8  $(2700^{\circ} \text{ C}, 1 \text{ min})$  underwent complete destruction of one portion of the capsule wall. The originally even inner surface of the capsule appears to have been eroded to a smooth wavy surface (fig. 5) with the ridges and valleys running parallel to the axial direction of the capsule and fuel pellet.

From an examination of the posttest radiographs of capsules 4, 6, and 7 (fig. 6), the progression of the reaction between the UN fuel and the cladding with increasing temperature (and decreasing time) appears as (1) a thinning of the cladding, (2) a decrease in the diameter of the fuel, and (3) the presence of a meniscus (showing solidifed material which apparently was liquid at the test temperature) at the bottom of the capsule.

Metallographic examination of capsule 5 (10 min at 2500<sup>0</sup> C) showed the only area

of reaction observed at this temperature. This was at the overlap in the layers of tungsten liner (see fig. 7). Uranium was transported from the fuel to the overlap area where it penetrated into both the tungsten liner and the T-111 cladding. This penetration was very slight, and a ductility test indicated no apparent decrease in the ductility of the T-111. A part of the area where reaction was observed in capsule 5 is shown in the electron-micrograph of figure 8. This area is adjacent to the overlap area shown in figure 7. The uranium phase has penetrated the T-111 alloy cladding and separated a 0.0013-centimeter (0.0005-in.) layer of the T-111 from the rest of the wall. This uranium phase also is present on the fuel side of the tungsten liner as a thin 0.0003-centimeter (0.0001-in.) layer. No tungsten or tantalum was detected in the uranium phase. No uranium was detected in either the T-111 or the tungsten phases.

Capsule 7 (2600<sup>°</sup> C for 10 min) showed much more extensive reaction and damage than capsule 5 (2500<sup>°</sup> C for 10 min). (See figs. 7 to 9.) Penetration of the grain boundaries of the T-111 can be seen in the figures: Dissolution of both the tungsten liner and the T-111 cladding are evidenced by the uniform thinning of the liner and the capsule wall. Fuel grain growth and a reduction of porosity with grain boundary widening are at and near fuel surfaces not in contact with the capsule walls. The large void area between the fuel and the capsule cladding (fig. 10) was occupied by fuel before the test. The void indicates that fuel has been lost from the fuel pellet. Fine precipitates, identified by scanning electron microscopy (SEM) as tungsten and tantalum, are visible at or near all areas of reaction where solidified free uranium was formed. At temperature. liquid uranium apparently collected either at the bottom of the capsule or where surface tension forces held it. On cooling, the uranium solidified and the meniscus at wetted surfaces was preserved. Figures 7 to 10 are typical of the microstructure of capsule parts in those capsules where temperature-time induced changes occurred. Figure 11 shows an SEM scan of an area of more severe reaction in capsule 7 ( $2600^{\circ}$  C, 10 min). The SEM spectrometer results indicate the following: Area A is a uranium phase with a trace of tungsten; area B is a phase consisting of tungsten, tantalum, and uranium; and area C is a uranium phase containing a trace of tungsten.

Analyses of cladding samples from the tests were made with an inert-gas fusion analyzer. Results of these analyses also are tabulated in table II. Cladding analyses were discontinued in those capsules where extensive reactions occurred. The presence of finely divided UN particles along the inner surface of the cladding resulted in misleadingly high nitrogen analyses for these claddings.

# DISCUSSION

# Causes of Observed Reactions

The tungsten that is used to eliminate the uranium mononitride - T-111 incompatibility problem cannot be regarded as a sealed lining because the end washers are loose and because tungsten is brittle and can develop cracks. At high temperatures, UN will dissociate and vaporize into free nitrogen and free uranium. Equilibrium pressures of nitrogen and uranium at the temperatures of interest are shown in table IV (refs. 8 and 9). Uranium mononitride vaporizes congruently up to a temperature of about 1800<sup>o</sup> C (ref. 10). In congruent vaporization two moles of free uranium are vaporized for every mole of nitrogen produced. By this mechanism UN can be brought into contact with the T-111 through discontinuities or cracks in the liners. If, over a period of time, a significant amount of UN is transported to the cladding, and/or nitrogen is gettered by the cladding (to form tantalum and/or hafnium nitrides) or permeates through the cladding, significant amounts of free uranium will be left inside the fuel pin. At temperatures above 1800<sup>0</sup> C, nitrogen is lost preferentially from the fuel (ref. 8). This results in the formation of a two-phase fuel consisting of UN and liquid uranium. If the liquid uranium. If the liquid uranium that is formed in the fuel at temperatures above  $1800^{\circ}$  C were to accumulate at the surface of the fuel, the protective function of the tungsten lining would be lost because of wetting and penetration of tungsten by the molten uranium (ref. 3).

Figure 8 shows that fuel-liner and fuel-cladding reactions did occur. The most severe attack is at the top and bottom of the fuel pellet where the pellet touched the tungsten capsule wall liner. The fuel, liner, and cladding reacted at these points with the reaction products of tantalum, tungsten, and uranium remaining. Varying degrees of damage occurred to the whole inner surface of the capsule. The surface grains of the fuel pellet densified and doubled in size. The grain boundaries of these surface grains widened, and a second-phase material was observed in these widened boundaries. This second phase was identified as free uranium.

From the nature of the attack (shown in fig. 9), it is deduced that decomposition of the fuel pellet occurred at the pellet surfaces. Nitrogen was released from the fuel pin leaving free uranium on the surfaces of the pellet. (Ref. 10 reports the presence of free uranium at the surface of UN which had been heated under vacuum at  $1835^{\circ}$  C.) The free uranium then apparently wet the tungsten liner and the tungsten-rhenium end spacers where they contacted the fuel pellet. As the liquid uranium spread, it penetrated all liner cracks, the liner overlap, and the liner-cladding interface. Thus, when the dissociation of the fuel pellet above  $1800^{\circ}$  C produces sufficient free uranium, the entire interior surface of the capsule can be wetted and damaged by the liquid uranium. In an actual reactor, the fuel also would be redistributed in the fuel pin by this mecha-

nism and could cause excessive reactivity changes.

The degree of capsule and liner attack by liquid uranium is dependent on the extent of decomposition of the fuel. If the nitrogen produced by the decomposition could be completely retained within the capsule (with a small internal free volume), little if any free uranium would be produced. The duration that the fueled capsule can remain at a specific temperature before cladding damage occurs is therefore dependent on the rate of loss of nitrogen from the capsule (or combination with the T-111 capsule wall) at that temperature. The results of this investigation show that the rate of loss of nitrogen from the capsule at temperatures of  $2500^{\circ}$  C and above is of sufficient magnitude to permit the formation of enough free uranium to damage the cladding in 1 hour at  $2500^{\circ}$  C and in less than 1 minute at  $2700^{\circ}$  C.

The nitride stability test results presented in table III show that appreciable nitrogen is released from the nitrogen-doped T-111 samples at temperatures of  $2500^{\circ}$  C and above in 10 minutes or less. And the T-111 can retain nitrogen in the form of nitrides only at a level well below the 500 ppm by weight, which was found necessary to cause embrittlement of the alloy (ref. 11). Most of the nitrogen released from the fuel must have been lost by permeation through the cladding. For temperatures above  $1800^{\circ}$  C, the information from this investigation indicates that nitrogen embrittlement of the fuel pin cladding will not occur. But in an actual reactor nitrogen released from the fuel pin probably would be gettered by T-111 in cooler parts of the reactor coolant loop. This potential problem is not considered in this report.

The phase diagrams for the uranium-tantalum (fig. 12(a)) system and the uraniumtungsten (fig. 12(b)) system indicate solubility of tantalum and tungsten in liquid uranium (ref. 12). As the capsule cools the tungsten and tantalum precipitate in the uranium.

#### Recommendations

The reactor program was cancelled before this work could be extended to obtain more detailed results on time-temperature dependence of fuel pin behavior. Important areas to be investigated to produce the information needed for preparing a detailed safety analysis for the reactor include the following:

(1) The rate of loss of nitrogen (and therefore the rate of accumulation of free uranium in the fuel) should be determined quantitatively as a function of temperature and time. This would permit the determination of allowable accumulated residence time for the reactor fuel pins at various temperature levels.

(2) The rate determining step in the mechanism of nitrogen transfer (e.g., dissociation, cladding nitriding, and nitrogen release to the vacuum from the cladding) should be investigated to see if there is a possibility of retarding the escape of nitrogen from the fuel pin.  $\cdot$ 

(3) The effect (if any) of fuel-pin scale-up and geometry on the temperature dependence of uranium accumulation rate in the fuel pin should be investigated. Fuel surfaceto-volume ratios, void fractions, and cladding thickness may affect the rate.

(4) Fuel redistribution during high-temperature excursions could cause the release of retained fission gases from irradiated fuel and change the stresses on the fuel pin cladding. The consequences of this possibility should be investigated in in-reactor studies.

In reference 4 a limit of  $1400^{\circ}$  C was set for transient temperature excursions in the reactor fuel pins. A number of emergency cooling systems were proposed to prevent this temperature from being exceeded in the event of a loss of coolant malfunction. From the results of this examination, this temperature limitation probably can be increased by about  $1000^{\circ}$  C, resulting in a possible weight savings in the design of the emergency cooling system.

## CONCLUSIONS

An out-of-pile test program was completed using simulated uranium mononitride fuel pins clad with tungsten-lined T-111 alloy to explore fuel stability and fuel-cladding compatibility as a function of time at temperatures well above the design concept reactor operating temperatures. Analyses of the test pins had led to the following major conclusions:

1. No cladding damage, fuel redistribution, or fuel dissociation was observed in test capsules heated to temperatures up to  $2400^{\circ}$  C for times as long as 8 hours. In view of the results of this investigation, no deleterious effects would be expected from occasional temperature excursions to  $2400^{\circ}$  C for an accumulated time of 8 hours during the normal lifetime of the reactor.

2. Fuel redistribution will occur at temperatures as low as  $2520^{\circ}$  C for a test time of 1 hour and in shorter times at higher temperatures. This redistribution is caused by the dissociation of the fuel into free uranium and nitrogen. Observable amounts of free uranium accumulate because of the loss of nitrogen by permeation through the tungsten liner and the cladding. The fuel, in the form of free uranium, will redistribute both by vapor transport and liquid transport and penetrate the tungsten liner and T-111.

Lewis Research Center,

National Aeronautics and Space Administration, Cleveland, Ohio, October 3, 1973, 503-25.

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| Element    | Co      | mposition     |  |  |
|------------|---------|---------------|--|--|
|            | wt %    | ppm by weight |  |  |
| Tantalum   | Balance |               |  |  |
| Tungsten   | 7.55    |               |  |  |
| Hafnium    | 2.46    |               |  |  |
| Cobalt     |         | 10            |  |  |
| Niobium    |         | 250           |  |  |
| Zirconium  |         | 500           |  |  |
| Vanadium   |         | 4             |  |  |
| Nickel     |         | 1             |  |  |
| Chromium   |         | 1             |  |  |
| Molybdenum |         | 20            |  |  |
| Iron       |         | 1             |  |  |
| Carbon     |         | 23            |  |  |
| Oxygen     |         | 30            |  |  |
| Hydrogen   |         | 4             |  |  |
| Nitrogen   |         | 25            |  |  |

### TABLE I. - CLADDING ANALYSIS

TABLE II. - SIMULATED FUEL PIN TEMPERATURE EXCURSION TESTS

| Capsule | Test         | Time. | Increase in                   | Results                                     |
|---------|--------------|-------|-------------------------------|---|
| number  | temperature, | min   | nitrogen content              |   |
|         | °C           |       | during exposure, <sup>a</sup> |   |
|         |              |       | ppm by weight                 |   |
| 1       | 1800         | 10    | 2.0                           | No detectable reactions; cladding ductile   |
| 2       | 2050         | 10    | 2.0                           | No detectable reactions; cladding ductile   |
| 3       | 2400         | 10    | 56 - 144                      | No detectable reactions; cladding ductile   |
| 4       | 2400         | 480   |                               | No detectable reactions; cladding ductile   |
| 5       | 2500         | 10    | 335                           | Very slight reaction; cladding ductile      |
| 6       | 2520         | 60    |                               | Small amount of reaction observed           |
| 7       | 2600         | 10    | (b)                           | Both fuel and cladding erosion observed;    |
|         |              |       |                               | cladding fractured during disassembly       |
| 8       | 2700         | 1     | (b)                           | Test scheduled for 10 min, but catastrophic |
|         |              |       |                               | failure of capsule terminated test after    |
|         |              |       |                               | 1 min; capsule cladding eroded through      |

<sup>a</sup>Inert gas fusion analysis. Sensitivity for nitrogen is  $\pm 0.5$  %.

<sup>b</sup>Value meaningless because of UN grains imbedded in cladding specimens.

#### TABLE III. - NITROGEN AND OXYGEN LOSS IN NITROGEN-DOPED

| Sample<br>number | Temperature,<br><sup>O</sup> C | Time,<br>min | Pretest interstitial content,<br>ppm by weight |        | Posttest interstitial content,<br>ppm by weight |        |
|------------------|--------------------------------|--------------|--|--------|---|--------|
|                  |                                |              | Nitrogen                                       | Oxygen | Nitrogen  | Oxygen |
| 1                | 2500                           | 10           | 1145   | 19     | 305   | 8      |
| 2                | 2500                           |              | 945  | 30     | 295   | 7      |
| 3                | 2600                           |              | 1150   | 16     | 420   | 6      |
| 4                | 2600                           | ↓ ↓          | 540  | 16     | 159   | 5      |
| 5                | 2700                           | 1            | 540  | 18     | 118   | 5      |
| 6                | 2700                           | 1            | 546  | 8      | 253   | 5      |

#### T-111 SAMPLES HEATED IN VACUUM



Figure 1. - Advanced power reactor concept fuel pin.



Figure 2. - Microstructure of as fabricated 95 percent dense UN fuel. X 500.





Figure 5. - Damaged test capsule after heating to 2700°C for 1 minute.



(a) Untested capsule.

(b) Capsule 4; heated 8 hours at 2400° C. (c) Capsule 6; heated 1 hour at  $2520^{\circ}$  C.

(d) Capsule; heated 10 minutes at 2600  $^{\circ}$  C.

Figure 6. - Radiographs of test capsules showing effect of increasing temperature and time on fuel pin damage.



Figure 7. - Fuel transport into tungsten liner overlap. Capsule heated to  $2500\,^{\circ}\,\text{C}$  for 10 minutes. X 500.



Figure 8. - Reaction area in capsule 5 heated to  $2500^{\,\circ}$  C for 10 minutes. Scanning electron micrograph;  $20^{\,\circ}$  tilt. X1000.



Figure 9. - Longitudinal section of fuel capsule 7 (heated to 2600° C for 10 min.) showing reaction areas and force uranium with precipitated tungsten and tantalum. Photomicrograph magnification, X125.



Figure 10. - Fuel-cladding reaction resulting from heating capsule to 2600° C for 10 minutes. The central part of the fuel is not shown. X50.



Figure 11. - Reaction area resulting from holding capsule 7 at 2000° C for 10 minutes. Scanning electron micrograph; 20° tilt. X1000.



Figure 12, - Phase diagrams (from ref. 12).