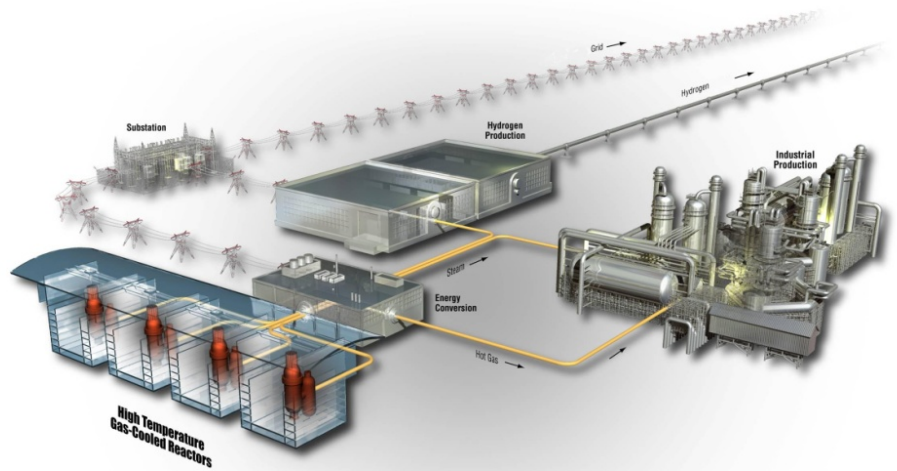


## Plan

Project No. 23841, 29412

# Research Plan for Moisture and Air Ingress Experiments

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## SUMMARY

Experiments to measure the oxidation of fuel element graphite and fuel matrix (both of NGNP specification) under moisture ingress and air ingress conditions are described. In the case of moisture ingress, the oxidation will take place under pressurized loss of coolant flow conditions, whereas under air ingress conditions, oxidation will take place under depressurized coolant conditions. Two types of experiments are proposed: one in which the oxidizing gas (either a water vapor – helium mixture or an air – helium mixture) is flowing through a mockup of a graphite fuel element with a stack of unirradiated compacts containing surrogate fuel particles, and the second in which the oxidizing gas is flowing over an irradiated fuel compact. Test requirements and preliminary test matrices are proposed with the recommendation that final requirements and test matrices be developed based on analyses with appropriate codes and informed by reactor design details to be forthcoming.

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## ACRONYMS

AGR	Advanced Gas Reactor
DOE	Department of Energy
DTF	designed-to-fail
FIMA	fissions per initial metal atom
HTTR	High Temperature Test Reactor
IAEA	International Atomic Energy Agency
MHTGR	modular high temperature gas-cooled reactor
NGNP	Next Generation Nuclear Plant
PSID	preliminary safety information document
R/B	release to birth ratio
TRISO	tristructural isotropic
UCO	uranium oxycarbide

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## 1. INTRODUCTION

The chemistry within the core of a high temperature gas-cooled reactor is highly reducing and stable during normal operation and under most off-normal and postulated accident conditions because of the overwhelming presence of many graphitic components and the inert helium coolant. There are, however, opportunities for the introduction of oxidants in the form of moisture and air under certain circumstances. Trace quantities of moisture, carbon monoxide and carbon dioxide may be present as impurities in the coolant during normal operation. The effects of long-term exposure of core materials, including fuel particles with both intact and failed coatings, to such impurities under reactor operating conditions are being investigated in the ongoing Advanced Gas Reactor (AGR)-3/4 irradiation test. Large amounts of moisture can be introduced into the core as a result of a leak in the steam generator of plants with a steam generator in the primary cooling loop, and significant amounts of air can be introduced following depressurization of the helium cooling loop in some accident scenarios. Research required to quantify the effects of the introduction of these relatively large ingresses of oxidants into the core during accidents is the subject of this research plan. Only a general guide to test conditions is provided. Detailed test matrices need to be developed based on analytical studies and initial test results. The status of understanding the effects of moisture and air ingress is discussed followed by the need for additional research.

## 2. MOISTURE INGRESS

The results of research on moisture ingress conducted prior to the initiation of the AGR program have been recently reviewed [Kendall and Hobbins, 2009] and a recommendation was made for fuel irradiation with helium coolant containing impurities. That recommendation is being carried out in the AGR-3/4 experiment and will also be addressed in the AGR-5/6/7 irradiation. Key results of the previous research are:

1. Hydrolysis of uranium oxycarbide (UCO) fuel affects only fuel particles with exposed kernels;
2. The time signatures of fission-gas release from hydrolysis are very similar in measurements taken in-pile and in post-irradiation heatup tests, as shown in Figures 1 and 2; and
3. The fractional release of stored fission gas from hydrolysis is a function of the partial pressure of the water vapor, as shown in Figure 3.

More recently investigation [Hobbins, 2010] of previous analyses of moisture ingress events in the modular high temperature gas-cooled reactor (MHTGR) Preliminary Safety Information Document (PSID) [DOE, 1986] has revealed that the reactor power is shutdown automatically and very quickly following the postulated moisture ingress. This is so that fuel hydrolysis occurs during post-irradiation heatup under pressurized loss of forced cooling conditions. While these analyses are design specific, the results provide a reasonable starting point for planning experiments on the effects of moisture ingress. Additional recommendations from the review of moisture ingress [Kendall and Hobbins, 2009] are to:



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- (a) Confirm that Next Generation Nuclear Plant (NGNP) UCO tristructural-isotropic (TRISO) fuel performance under hydrolysis conditions is equivalent to that observed in previous experiments;
- (b) Generate experimental data under post-irradiation testing to moisture levels enveloping the maximum water partial pressure predicted for the NGNP;
- (c) Measure metallic fission product release under moisture ingress conditions;
- (d) Perform a systematic review of current computer codes and methods for modeling the effects of moisture ingress; and
- (e) Perform scoping calculations to better define conditions for the proposed experiments.

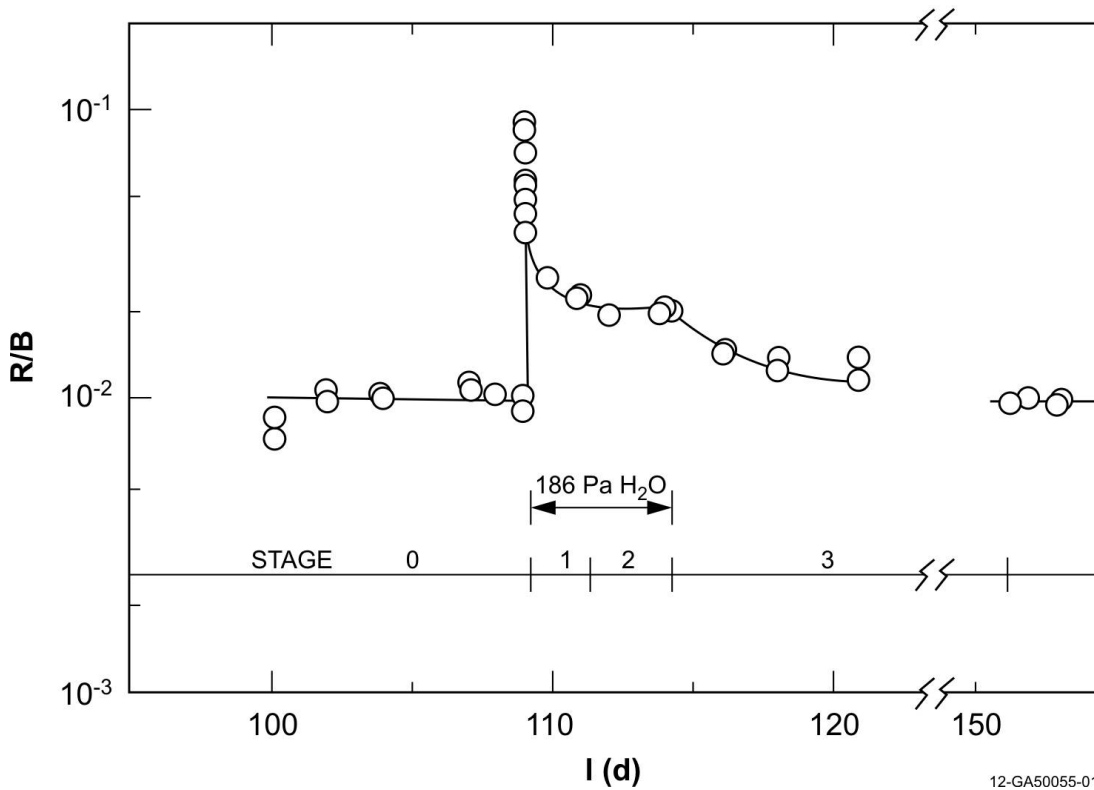
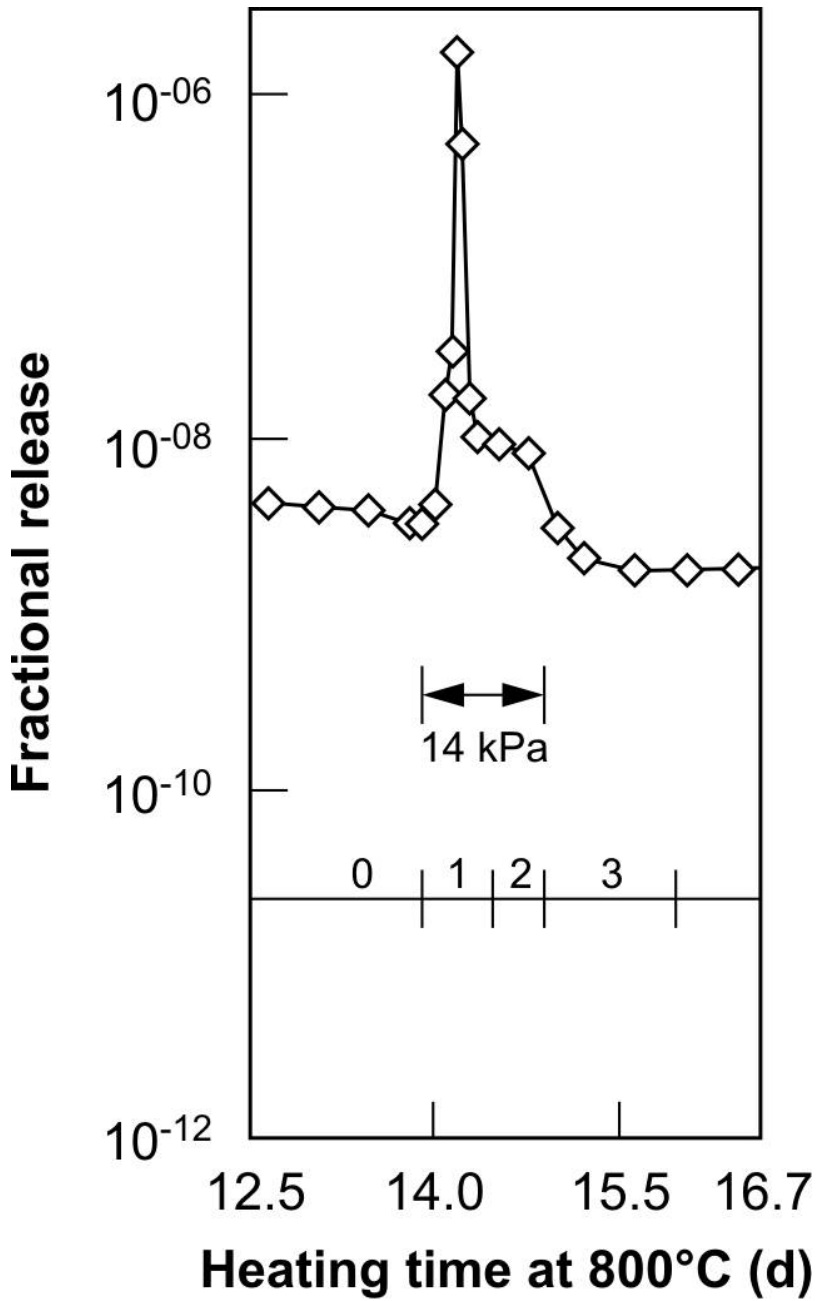


Figure 1. Release to birth ratio (R/B) time profile of Kr-85m for water injection of 186 Pa at 767°C for UCO fuel compacts with DTF particles containing only UO<sub>2</sub> phase [Myers, 1995].

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Figure 2. Fractional release rate ( $s^{-1}$ ) of Kr-85 during 24-hour water injection under post-irradiation heating at 800°C of UCO fuel containing only UO<sub>2</sub> phase [Myers, 1995].

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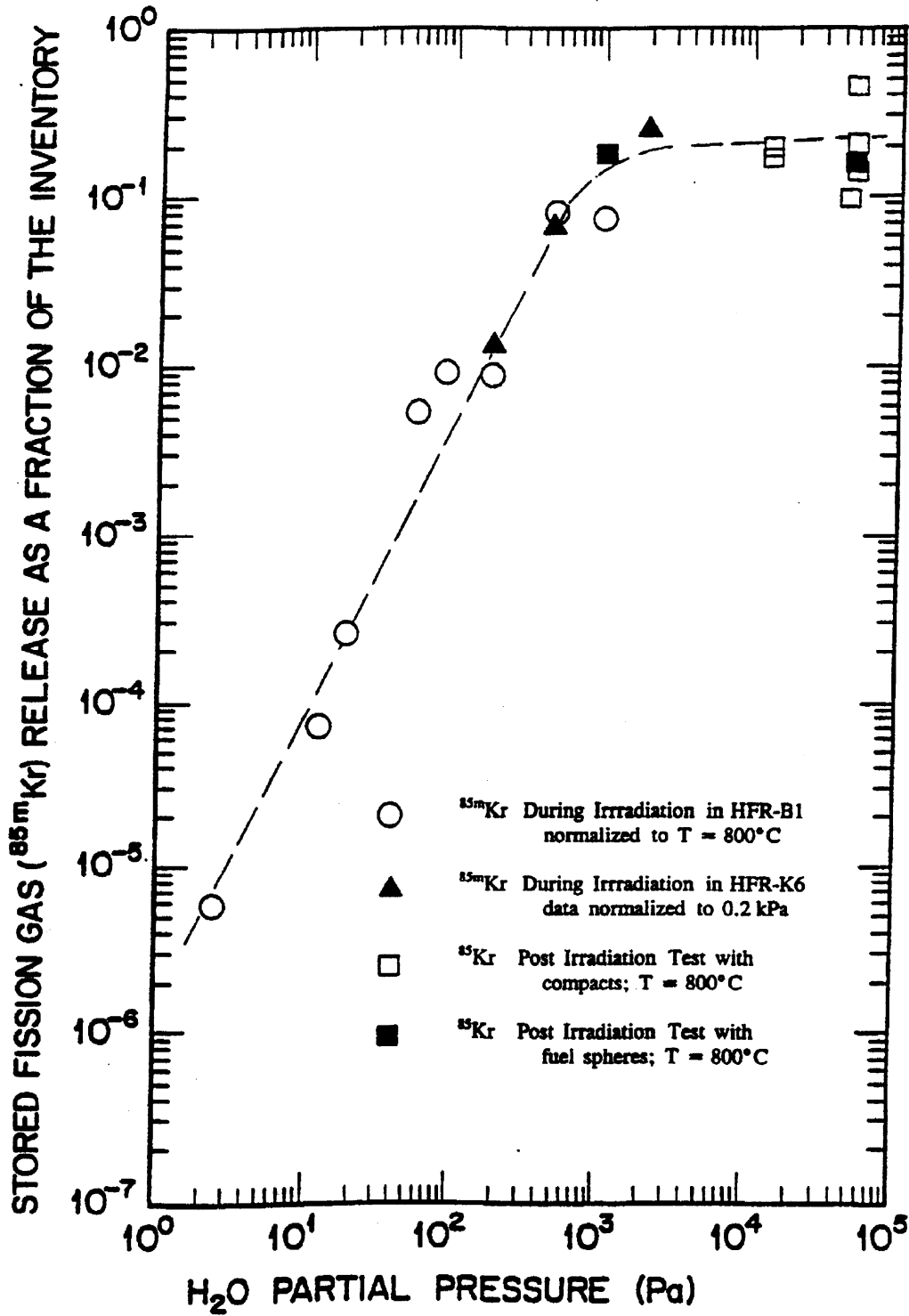


Figure 3. Fractional release of fission gas as a function of partial pressure of water vapor [Myers, 1995].

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This research plan is targeted on the effects of oxidants on the core; however the need for additional work on radionuclide removal from primary surfaces under wet depressurization conditions may need to be reviewed, as mentioned in [Kendall and Hobbins, 2009].

## 2.1 Proposed Experiments

Two types of experiments, both out-of-pile, are proposed: one using irradiated fuel compacts containing a known fraction of designed-to-fail UCO particles, with some of the compacts enclosed in a graphite body; and the other using unirradiated fuel compacts, containing intact surrogate ( $ZrO_2$ ) fuel particles, in a mockup of a graphite fuel element. An early assessment of the number of compacts of both types envisioned for the experiments versus availability of both types of compacts, including the burnup and fluence of irradiated compacts, will be needed. The experiment with irradiated fuel particles will be designed to satisfy recommendations (a), (b), (c), (d) and (e) above. The experiment with surrogate fuel particles will be designed to measure the oxidation of graphite surrounding the fuel compacts, the oxidation of the fuel compact matrix, and attack of the SiC layer of the fuel particles. This experiment was proposed earlier [Montgomery, 1987] to enhance limited databases on the rate of oxidation of graphite and compact fuel matrix in high partial pressures of water vapor under accident conditions and to give confidence to predictions made by the OXIDE computer code [Peroomian, et al., 1974]. Comparison of the results of the two types of proposed tests will elucidate reaction rates of moisture with irradiated and unirradiated graphite and matrix materials. Experiments such as the two proposed here, which include fuel compacts enclosed in graphite exposed to flowing helium containing water vapor, require the water vapor to be transported through the graphite before it can react with the compact matrix. Calculations with appropriate analytical methods, such as OXIDE, REACT [Richards, et al, 1990 (1)] or other codes should be performed to make predictions of graphite and matrix oxidation and fuel hydrolysis to guide finalizing experimental conditions and refine the test matrix.

Review of design basis events and safety-related design conditions analyzed in the MHTGR PSID [DOE, 1986] indicates that reaction of moisture with core materials is limited as a result of termination of ingress due to isolation of the steam generator or venting of the steam due to depressurization upon automatic opening and subsequent failing open of the pressure relief valve (however, this is design dependent). In the postulated scenarios, much of the steam has been vented by the time the core temperature has reached  $1250^{\circ}C$  [Montgomery, 1987]. Analysis of SRDC-6, a depressurized conduction cooldown with moderate moisture ingress, indicates the time to reach a maximum core temperature of  $1250^{\circ}C$  to be about 6.5 hours, as shown in Figure 4 taken from [DOE, 1986]. In their review of [DOE, 1986] NRC staff requested additional analyses with longer times before the failure of the pressure relief valve, which resulted in higher doses. The results of testing will need to address conditions that bound events identified in the licensing review. Although water partial pressures as high as 3.5 atm could be expected in MHTGR accident scenarios [Richards, et al., 1990(2)], calculations show that above about 0.3 atm partial pressure of water vapor, the rate of grade H-451 graphite oxidation is independent of steam pressure [Montgomery, 1987]. In addition, the data on fuel hydrolysis in Figure 3 indicate that the fractional release of fission gas saturates at a water -vapor partial pressure of about 0.1 atm. These results suggest that testing conducted at atmospheric pressure with water vapor partial pressures less than 0.5 atm will be sufficient to elucidate the behavior expected in the reactor.

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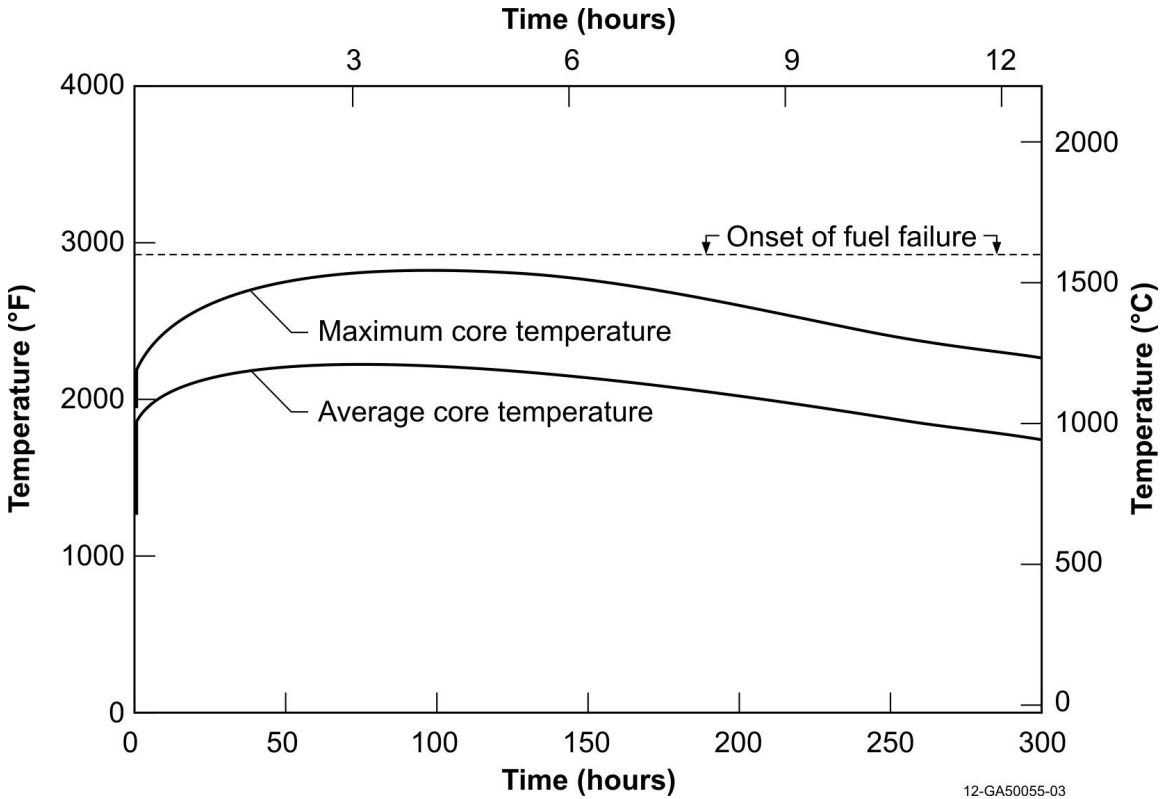


Figure 4. Core temperature evolution in SRDC-6 [DOE, 1986]. This is a generic maximum time-temperature profile useful for planning purposes.

This conceptual plan is based on initially isothermal tests with gas inputs at constant composition. This approach simplifies analysis of experimental results and is useful for comparison with code results. Later tests under more realistic transient conditions, including time variations of inlet gas composition and test temperature, may be useful for examining various accident scenarios.

### 2.1.1 Experiments with Compacts of Surrogate Fuel Particles in a Fuel Element Mockup

A series of oxidation experiments with compacts of surrogate fuel particles (TRISO-coated ZrO<sub>2</sub> kernels) contained in a mockup of a graphite fuel element (of composition selected for the NGNP) is proposed, similar to that previously proposed in [Montgomery, 1987]. The fuel element mockup features a stack of fuel compacts surrounded by a triangular array of coolant holes through which helium containing water vapor flows, as shown in Figure 5. The purpose of these experiments is to measure the rates of oxidation of the graphite and the matrix and to determine the extent of attack of the SiC layer in the TRISO fuel particles in a prototypic geometry. Other geometries may be selected based on the results of calculations, separate effects testing, and reactor design information.

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Following the recommendation in [Montgomery, 1987], it is proposed to conduct experiments at 1000, 1100 and 1250°C, as these are reasonable starting points in the absence of specific design dependent accident scenarios. Rather than using only one gas composition (He plus 0.3 atm water vapor) as specified in [Montgomery, 1987], it is suggested to use a range of water vapor partial pressures entering the test chamber, such as 0.1, 0.2, 0.3, and 0.4 atm, to cover a range of moisture ingress conditions from small to large and to confirm the prediction that the rate of oxidation of graphite (of NGNP composition) saturates at about 0.3 atm. After each test, the fuel compacts should be removed from the graphite fuel element mockup and the compacts and the fuel element weighed individually to measure weight loss (fraction burnoff). The compacts should be cross-sectioned and examined by metallography to document any evidence of attack of the SiC layer on the surrogate fuel particles. Since this is an out-of-pile test, additional options for post-test examinations, such as scanning electron microscopy among others, could be considered.

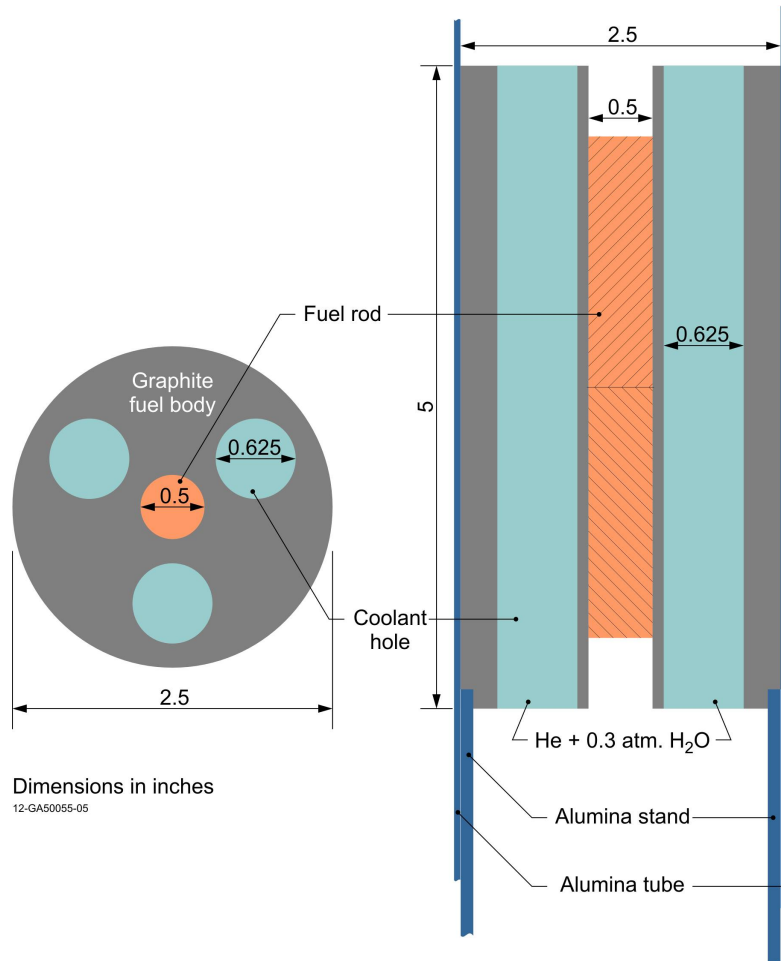


Figure 5. Schematic showing integral oxidation test geometry in graphite fuel element mockup [Montgomery, 1987]. The experiment design will be finalized based on more detailed analysis.

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If moisture is able to penetrate the OPyC layer, reaction with SiC would be expected to form SiO<sub>2</sub> at 1250°C at local water vapor pressures above 0.05 atm (the passive region), see Figure 6 taken from [Minato and Fukuda, 1993]. At lower water vapor partial pressures at this temperature, in the active region, volatile SiO is expected and thinning of the SiC layer should be evident. At lower temperatures, Figure 6 indicates the transition from the passive to active region occurs at lower water partial pressures. Experiments in the active as well as the passive region are recommended to verify the transition parameters. If deemed important for understanding accident consequences, small slabs of CVD SiC could be exposed directly to low steam partial pressures to collect degradation data.

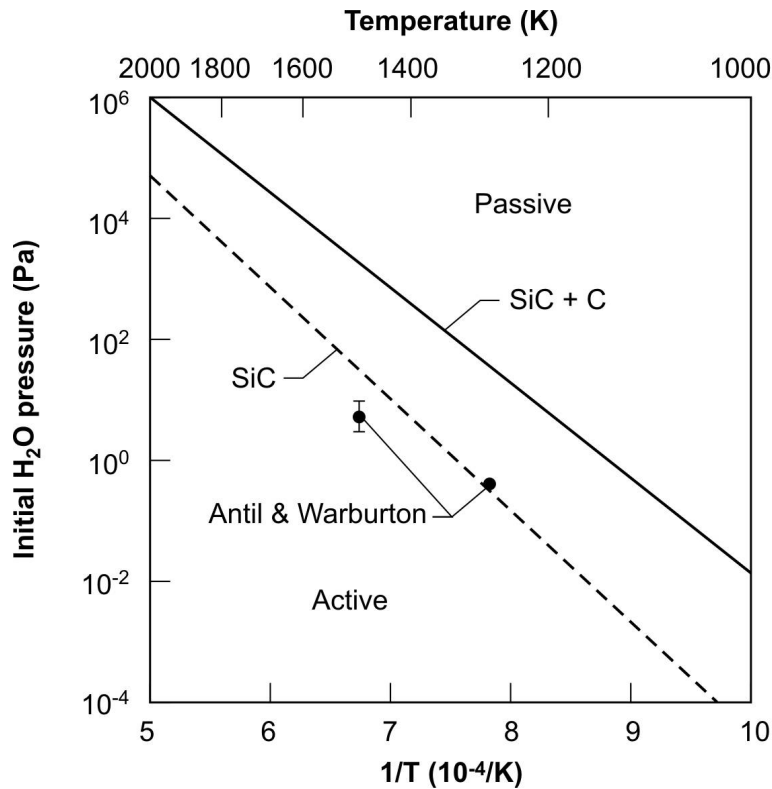


Figure 6. Active to passive oxidation transitions for SiC and SiC + C for water ingress [Minato and Fukuda, 1993].

Although water vapor at high partial pressure is available only for a limited time with this postulated accident, no more than about 10 hours according to analysis of SRDC-6, it could be longer for different event sequences, and it was originally proposed to continue these experiments for longer times, if necessary, to achieve nearly 100% burnoff of the matrix [Montgomery, 1987]. Test durations up to 24 hours could be considered to be consistent with post-irradiation heatup tests described below. (Note that test duration may differ from a particular accident scenario because of the desire to obtain data for use in codes rather than simply simulate an off-normal event.) A gas flow rate of approximately 80 L/min of helium containing 1% hydrogen plus water vapor was suggested in [Montgomery, 1987]. The hydrogen in the inlet gas mixture is added to insure that the concentration of hydrogen is constant at the local level throughout the test specimen. More than one level of hydrogen concentration should be considered. Testing of

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matrix only compacts (which are available) at low partial pressures of moisture in an existing graphite oxidation apparatus to measure reaction rate kinetics should be considered. These recommendations should be revisited in the development of the detailed test matrix, which will depend on the needs of codes and on the reactor design.

An important and complicating factor in these experiments is that at these accident temperatures water vapor reacts with, but does not readily transport through, graphite, resulting in water -vapor partial pressures at the surface of the fuel compact much lower than that flowing in the coolant channels [Richards, et al, 1990 (1) and Myers, 1991]. In addition, the fuel matrix for the NGNP may be more reactive with water vapor than is graphite, but there are no data for matrix oxidation above 950°C [Montgomery, 1987]. As a result of these uncertainties, a range of partial pressures (0.1 to 0.4 atm) as proposed is appropriate. It should also be recognized that the test matrix might require changes after analysis of the results of initial testing.

Research on the corrosion of SiC under high water-vapor pressures has been conducted to understand environmental effects on SiC-based composites for gas turbines [Opila and Hann, 1997, Opila, 1999, and More, et al., 2000]. These studies indicate that the corrosion rate of SiC increases with H<sub>2</sub>O partial pressure such that at 1200°C the time to corrode completely though the 35 µm SiC layer in a TRISO fuel particle would be predicted to decrease from 15,000 hours at 0.9 atm to 390 hours at 1.5 atm. Even though water-vapor pressures up to 3.5 atm have been calculated for large water ingress accidents, the likely saturation of the reaction rate of graphite with water at about 0.3 atm, combined with the slow transport of moisture through the graphite and the limited duration of exposure to high pressure water vapor (less than 10 hours), suggest that intact SiC layers will likely be found in the TRISO fuel particles in the proposed tests.

### **2.1.2 Post-irradiation Heatup Experiments**

It is proposed to perform post-irradiation heatup experiments at conditions similar to those of the experiments with unirradiated fuel compacts discussed in Section 2.1.1: temperatures in the range 800 to 1250°C in flowing helium with the injection of water vapor at partial pressures between 0.01atm and 0.3 atm for a duration in the range of 10 hours (max expected) to 24 hours (to be consistent with previous tests). The test temperatures, water vapor partial pressures, and exposure durations will be determined as the reactor designers complete their analyses; however, the above values serve as baseline conditions to connect with past work and provide guidance for planning. Similar to the tests in Section 2.1.1, hydrogen additions to the inlet gas should be considered. Initial tests should be isothermal as the results are easier to interpret. Transient tests could also be considered. For example, given that moisture could more easily penetrate graphite at lower temperatures, a test starting at a lower temperature and increasing over time could be more limiting as well as more representative. The purpose of the experiments at 800°C is to provide a comparison with the previous results shown in Figure 3. Some of the irradiated fuel compacts should contain a known fraction of DTF particles. Some of the compacts should be tested within a closed graphite body (preferably irradiated). A source of such compacts is the AGR-5/6/7 irradiation experiment. Re-irradiation to permit the measurement of Xe-133 (as a surrogate for I-131) should be considered if a cost effective method is available. The compacts should be weighed posttest to determine the fraction burnoff and compacts should be sectioned and examined by metallography to determine the extent of attack of the SiC layer, if any.

The release of fission gases and metals should be measured as a function of time. Gases can probably be measured in a manner similar to that used in inert furnaces currently in operation for



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safety testing, but fission metals may be more difficult to collect in the oxidizing environment and new techniques may have to be developed. Fission metals will be released from three sources: oxidation of kernels and oxidation of matrix in compacts containing previously failed particles (such as DTF particles), and particle failures occurring under the post-irradiation testing. Measurements of fission gases will provide temporal information on releases from kernels since the matrix will not be a source of gases.

### 3. AIR INGRESS

Results of experiments using unirradiated compacts and fuel particles in Japan and irradiated fuel spheres and fuel particles in Germany have been reported [IAEA, 1997]. The Japanese results of weight change as a function of time indicate that oxidation of carbonaceous materials in an unirradiated fuel compact is complete after 20 hours in flowing air at 1400°C, revealing the SiC layer of the particles. After this duration, the particle failure fraction was determined to be  $6.9 \times 10^{-4}$ . After 54 hours in flowing air at 900°C, a particle failure fraction of  $1.2 \times 10^{-3}$  was measured. German results on irradiated spherical fuel elements (burnup of about 9% FIMA) indicated a particle failure fraction of  $2.4 \times 10^{-4}$  after 410 hours in flowing air at 1,300°C,  $7.3 \times 10^{-4}$  after 70 hours at 1,400°C and  $1.2 \times 10^{-3}$  after 140 hours at 1400°C. These limited results suggest that significant fuel failure can be expected after tens of hours in flowing air at temperatures in the range 1300 to 1400°C.

More recently as part of the NGNP program, the ingress of air into the reactor core following coolant depressurization has been comprehensively studied both analytically and experimentally [Oh, et. al., 2011, Oh and Kim, 2011, and Oh, et al, 2006]. A major finding has been that the onset of natural circulation of air through the core is rapid, on the order of 100 to 500 s, following complete depressurization. Natural circulation begins relatively quickly for a larger break, whereas for a smaller break the depressurization phase takes many hours before the system is completely depressurized and natural circulation can begin to occur. The flow rate through the core under natural circulation is in the range 0.1 to 0.2 m/s for all break sizes and locations because the principal driving force is the temperature difference between the core and the coolant riser [Kim, 2011]. These values are expected to hold for either a prismatic or pebble bed core [Schultz, 2012]. Note that these values do not take into account any mitigating safety systems, but are a useful starting point for purposes of planning experiments.

Oxidation of IG-110 graphite (the Japanese standard graphite used in the High Temperature Test Reactor [HTTR] reactor core) in air was measured using a gas concentration (CO and CO<sub>2</sub>) analysis method, stated to be faster and more precise than thermogravimetric analysis [Oh, et al., 2006]. The results shown in Figure 7 demonstrate that the oxidation rate increases with increasing oxygen concentration and saturates at temperatures of about 1200°C. The GAMMA code used in the analyses of air ingress phenomena [Oh, et. al., 2011, and Oh, et al. 2006] has been updated to include the results of these oxidation rates. In simulations of oxidation in a prismatic core, the reactor core was modeled as a porous medium. In order to use the GAMMA code for analysis of the effect of air ingress on fuel compacts, the code will need to be benchmarked by oxidation experiments in a more complex geometry representing the mockup of a prismatic fuel element [Kim, 2011]. Such an experimental setup was shown earlier in Figure 5.

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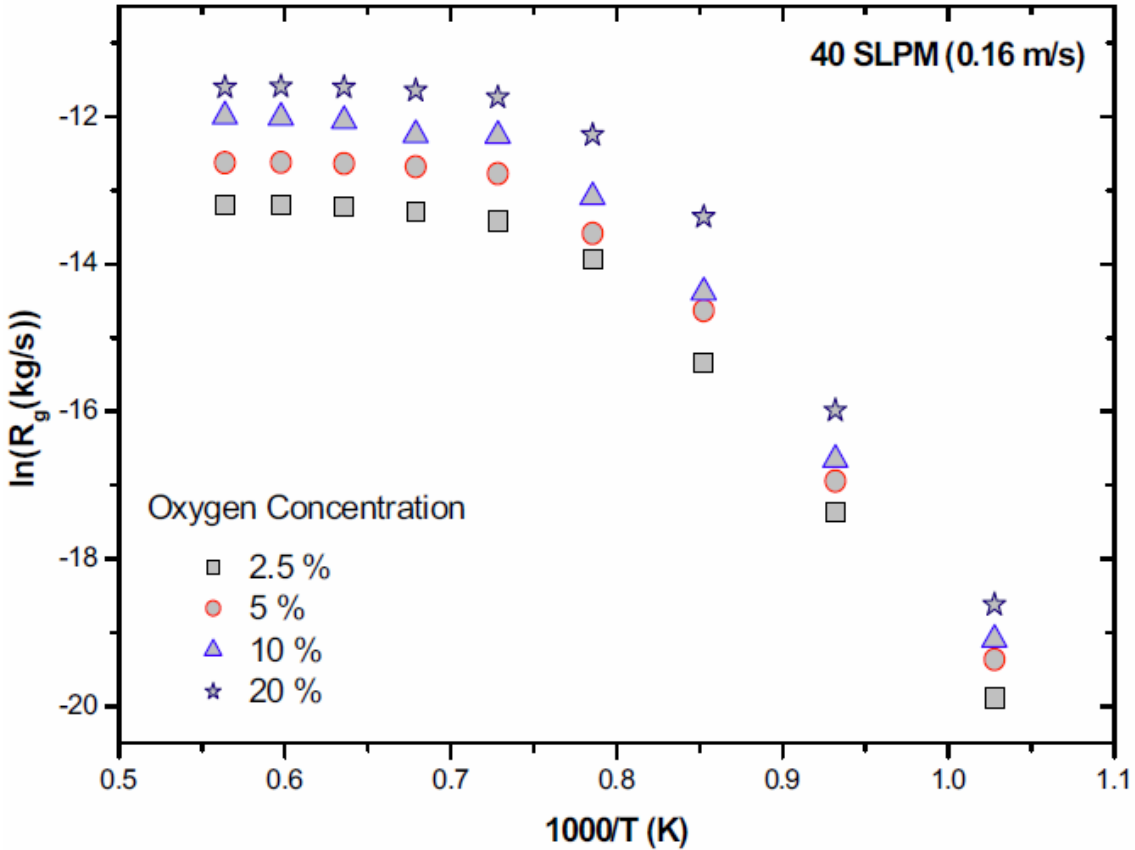


Figure 7. Rate of graphite oxidation in helium – oxygen mixtures [Oh, et al., 2006]

### 3.1 Proposed Experiments

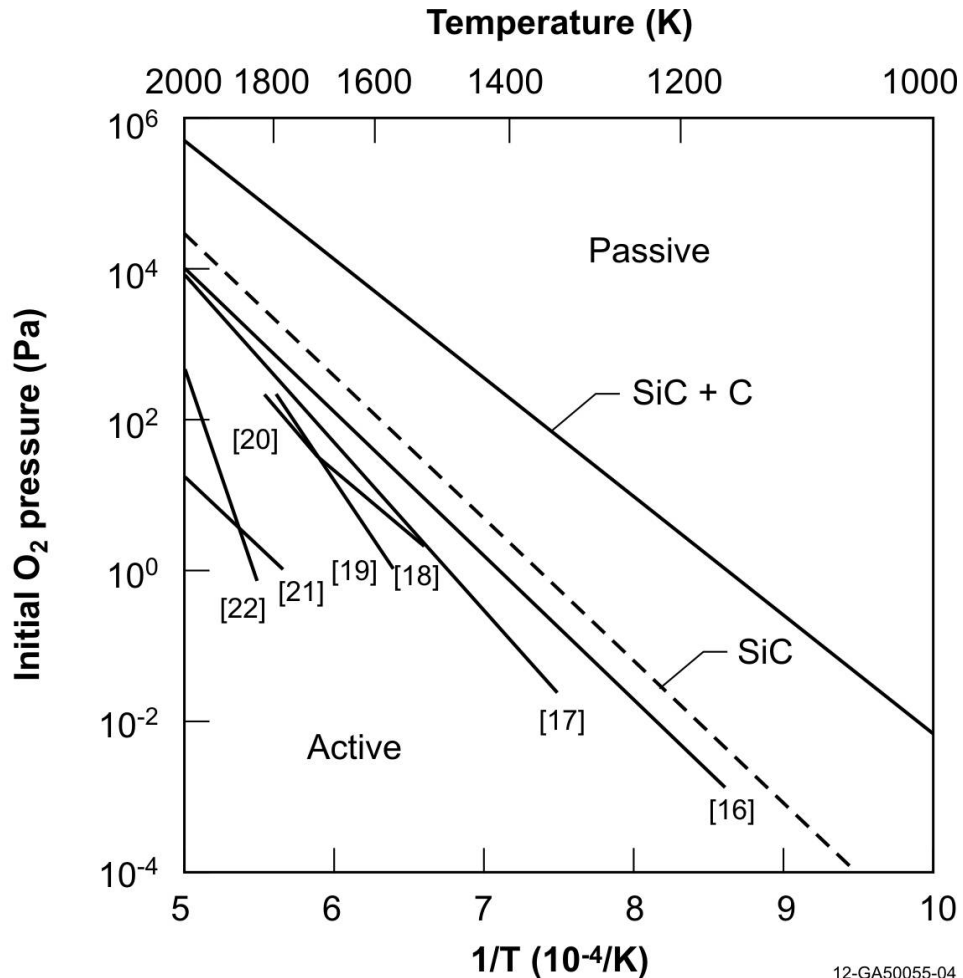
Experiments proposed for air ingress are of the same two types as those proposed for moisture ingress.

#### 3.1.1 Experiments with Compacts of Surrogate Fuel Particles in a Fuel Element Mockup

For experiments with compacts of surrogate (ZrO<sub>2</sub>) TRISO-coated fuel particles in a stack within the mockup of a prismatic fuel element, temperatures in the range 1000 to 1600°C (the maximum temperature for depressurized coolant accidents) in flowing helium containing air at several fractions are envisioned. Additions of CO and CO<sub>2</sub> to the inlet gas to set constant local conditions throughout the test specimen should be considered. Saturation of the oxidation rate at higher temperatures may obviate the need to test at the highest temperatures. The gas flow rate should be in the range 0.1 to 0.2 m/s to match natural circulation within the core. These values should be updated when new design analysis information is available. The duration of experiments should be dictated by the exposures required to fully characterize the oxidation as a function of temperature and oxygen concentration. As in the case of moisture ingress, initial experiments, for ease of interpretation, should be isothermal, but more representative transient

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tests could be considered. Development of the detailed test matrix, including fractions of air in helium and test durations, should be informed by analyses with codes such as GAMMA and OXIDE. Oxidation of the graphite (a composition to be used in the NGNP) and fuel matrix (NGNP specification) will need to be determined gravimetrically by measuring weight loss of each component separately. Attack of the SiC layer will be determined by metallographic examination of compact cross sections and by additional examination techniques, such as scanning electron microscopy among others, that may be appropriate for these unirradiated materials. The transition between regions of passive and active oxidation of the SiC based on a thermodynamic evaluation of air oxidation in the presence of graphite is shown in Figure 8 [Minato and Fukuda, 1993]. The range of oxygen partial pressures should cover both the passive and active oxidation regimes. As in the case of moisture ingress, separate tests to measure the oxidation kinetics of the compact matrix should be considered.



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Figure 8. Active to passive oxidation transitions for SiC and SiC + C for air ingress [Minato and Fukuda, 1993].

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**3.1.2 Post-irradiation Heatup Experiments**

Irradiated fuel compacts, some inside closed graphite bodies (irradiated preferred), should be heated to temperatures in the range 1000 to 1600°C in helium-air mixtures. As with the experiments in Section 3.1, addition of CO and CO<sub>2</sub> to the inlet gas should be considered. The releases of gaseous and metallic fission products should be measured as a function of time. As noted in Section 2.1.2, new techniques may be required to collect fission metals and understanding of temporal metal releases from compacts containing DTF particles may be complicated by releases from the matrix. Initial tests should be isothermal, but more representative, transient tests could also be considered. Durations of isothermal tests should be up to 100 hours or more to measure significant fractional fission product releases. While saturation of the oxidation rate at higher temperatures might obviate the need to test at the highest temperatures with respect to oxidation, the higher temperatures could lead to greater fission product release and thus should be considered. Potential sample materials include compacts containing DTF fuel particles, some in closed graphite bodies, from the AGR-5/6/7 experiment, and others without DTF fuel particles from the AGR-2 and AGR-5/6 irradiations. Re-irradiation to permit the measurement of Xe-133 (as a surrogate for I-131) should be considered. Formulation of a detailed test matrix should be informed by analysis with codes such as GAMMA, OXIDE and others, as appropriate.

**4. TESTING REQUIREMENTS**

Furnaces to heat specimens to the temperatures and in the envisioned oxidizing environments (mixtures of helium and moisture and helium and air) are required. The apparatus for experiments with compacts of unirradiated surrogate fuel compacts in the fuel element graphite mockup (laboratory) and the apparatus for post-irradiation heating of irradiated compacts (hot cell) must have the capability to monitor the composition of the gas entering the furnace and the gaseous reaction products exiting the furnace. In addition, the hot cell apparatus must have the capability to measure fission gases and fission metals (could be oxides) released from the fuel. The apparatuses should be designed to permit programmable temperatures and gas compositions.

A brief description of an apparatus (KORA) designed to heat irradiated fuel particles, fuel compacts and spherical fuel elements to temperatures up to 1600°C in flowing helium containing water vapor or air is given by [Schenk and Nabielek, 1993]. In practice, the apparatus was used only at 800°C and with helium containing up to 50 kPa (0.5 atm) water vapor. Only measurements of Kr-85 were made. The requirements of the apparatuses for the experiments envisioned in the current plan significantly exceed the operating experience of the KORA apparatus.

**5. PRELIMINARY TEST MATRICES**

Preliminary test matrices for moisture and air ingress experiments are presented in Table 1. Final, more detailed matrices need to be developed based on analytical studies with appropriate codes, such as OXIDE, REACT, GAMMA, and initial test results.

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Table 1. Preliminary Text Matrices

Oxidant	Conditions	Unirradiated Surrogate Fuel	Irradiated Fuel
Water	Temperature	1000 - 1250°C	800 - 1250°C
	Gas Composition	He + up to 0.4 atm H <sub>2</sub> O	He + up to 0.3 atm H <sub>2</sub> O
	Gas Flow Rate	~ 80 L/min	~ 80 L/min
	Test Duration	10 – 24 hours	10 – 24 hours
Air	Temperature	1000 - 1600°C	1000 - 1600°C
	Gas Composition	He + air (Fract. TBD)	He + air (Fract. TBD)
	Gas Flow Rate	0.1 to 0.2 m/s	0.1 to 0.2 m/s
	Test Duration	~ 100 hours	~ 100 hours

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