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**LIGHT-WATER-REACTOR SAFETY
RESEARCH PROGRAM:
QUARTERLY PROGRESS REPORT**

January—March 1975

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

**Prepared for the U. S. NUCLEAR REGULATORY COMMISSION
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January-March 1975

Robert G. Sachs, Laboratory Director
Jack A. Kyger, Associate Laboratory Director

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FOREWORD

This report is the first in a series of quarterly progress reports covering work on water-reactor-safety heat-transfer and flow problems. The work reported here therefore covers time before the reporting period in order to provide the reader with sufficient background material relative to the current work.

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I. LOSS-OF-COOLANT ACCIDENT RESEARCH; HEAT TRANSFER AND FLUID DYNAMICS

P. A. Lottes, RAS

Research in heat transfer and fluid flow covers (1) analysis of PWR FLECHT-SET oscillations, (2) two-phase vessel-blowdown experiments, and (3) transient critical-heat-flux experiments.

The PWR FLECHT-SET analysis to date is an attempt to produce a simplified mathematical model to describe in a qualitative manner the results obtained in the FLECHT-SET experiments. One objective of the analysis is to provide insight on the nature of the refilling process during the quenching phase of a reactor core following a postulated blowdown. Results of the analysis are described here.

The two-phase vessel-blowdown experiments were performed to get fundamental information on critical discharge rates from a vessel during rapid depressurization. Measurements of transient mass flow rate, pressure, and temperature are described and discussed. The system behavior is interpreted from the transient measurements. The transient system pressure is shown to be influenced by the presence of surfaces containing nucleation sites. Data are compared to the Moody model, the Henry-Fauske model, and the homogeneous equilibrium model.

The transient critical-heat-flux experiment has been designed to produce fundamental information on the process of tube-wall overheating during a combined pressure and flow transient. As a result of the design criteria, extensive care has been used in the selection and calibration of instrumentation. The system is described in general terms. A few isothermal tests, as well as some steady-state critical-heat-flux tests, have been performed. Details of the test results and information regarding instrument calibrations will be included in the next quarterly report.

A. Analysis of PWR FLECHT-SET Oscillations (R. P. Stein, RAS)

The following is a summary of the main results of this study obtained to date. The results were obtained by analysis based on an idealized mathematical model of the FLECHT-SET Phase-A apparatus, and by examination of the available FLECHT-SET experimental data. The mathematical model was derived by treating the system as a U-tube manometer, with coolant injection into one of the vertical legs representing a PWR downcomer. The other vertical leg represents the core region and includes various formulations to account for pressure changes caused by boiling and flow through a simulated upper plenum. Initially, only uniform (one-dimensional) flow through the simulated core region was considered. The model was then extended to include multiparallel flow subchannels in the core region, with no "crossflow"

between subchannels, except at their common lower plenum. Current efforts are focused on attempts to extend the model further to include crossflows.

The results obtained pertain only to about the first 60 s or less during reflood, since oscillatory behavior in the FLECHT-SET experiments was most severe during this time period. Also, in most of the experiments the simulated core region appears to be completely flooded after this initial time period. As a result, oscillatory behavior is then strongly affected by liquid flows through and out of the simulated upper plenum and in many cases by overflows from the simulated downcomer.

The following results were obtained:

1. Large-amplitude oscillations of core-flooding velocities occur, even in the absence of pressure changes caused by boiling.
2. The detection of oscillatory flooding rates in the experiments depends heavily on attempting to infer oscillating downcomer liquid levels from pressure measurements at the bottom of the downcomer. It is extremely unlikely that oscillations of the type referred to in result 1 above can be detected in this manner, since the amplitude of the corresponding oscillatory downcomer pressure will be very small. Oscillatory downcomer pressures of significant amplitude occur only when there are corresponding oscillatory pressures in the simulated core region.
3. Damping, resulting from frictional dissipation, is small during the initial high-coolant-injection-rate period, and nearly negligible during the low-coolant-injection-rate period. However, this result depends on the use of traditional methods to estimate frictional losses, which could be highly uncertain in a system involving changes in flow direction, expansions and contractions in complicated configurations, and transients.
4. Attempts to duplicate by computation the type of oscillatory downcomer pressure response observed with the experiments were unsuccessful when such attempts were based on the use of simplified boiling pressure drop and simulated fuel-rod heat-transfer relationships to account for pressure changes caused by boiling in the simulated core region. Duplication of downcomer pressure response was fairly successful, although mostly in a qualitative sense, when based on purely parametric models to account for pressure changes caused by boiling. It was necessary that these parametric models simulate nearly instantaneous and relatively large pressure changes due to recurring boiling inception and quenching.
5. When the oscillatory downcomer pressure response observed with the experiments was duplicated by computation as described above, the amplitudes of the corresponding oscillatory flooding velocities were extremely large.

6. Computations with the model that included three nonconnected parallel subchannels in the simulated core region indicated that significantly nonuniform oscillatory behavior among the subchannels could result from nonuniform heat generation, if transversely nonuniform pressure changes caused by boiling were sufficiently large. Since transversely nonuniform pressures of this kind would be more likely to occur in a large core region of PWR size, and very unlikely in the small core size of the experiment, this result suggested a possible difference between experiment and prototype that could influence oscillatory behavior. In particular, nonuniform behavior among subchannels would favor the occurrence of significant crossflows, which could result in an additional mechanism for frictional dissipation not present in the experiment.

7. Computations with the three-parallel-subchannel core-region model mentioned above, which also included a crude and largely intuitive representation of the potential effects of crossflows among subchannels, indicate the existence of an optimum transverse flow resistance with respect to additional frictional dissipation caused by crossflows. When transverse flow resistances are sufficiently large, small decreases in this resistance result in an increase in system frictional dissipation. When transverse flow resistances are not sufficiently large, decreases in this resistance result in a decrease in system frictional dissipation. Because of the crude representation used to model the potential effects of crossflows, the results of these computations can only be considered as qualitative and suggestive. However, the same general effect has been obtained by analysis of a simpler, but more accurately formulated, model of a U-tube manometer with an additional variable-resistance cross-connect between vertical legs, but without coolant injection and without pressure changes caused by boiling.

B. Two-phase Vessel Blowdown (M. N. Hutcherson and R. E. Henry, RAS)

A study has been performed on the two-phase vessel-blowdown phenomenon with particular emphasis on investigating the critical flow phenomenon in the transient, large-duct case, and also the effect of internal vessel geometry upon the decompression. The intent was to investigate the characteristics of the depressurization while simulating a full-open, double-ended, guillotine break in the inlet coolant line of a light-water reactor which would force the descending fluid in the downcomer annulus and the ascending fluid in the core region to stagnate and then reverse flow direction in order to exit the break in the inlet line. The experimental apparatus used in this investigation (shown in Fig. I.1) simulated the downcomer geometry having an internal "skirt." The experimental model was about one-sixth the size of a currently operating pressurized-water-reactor design, as gauged by the ratio of the hydraulic diameter of the exit duct of the model to the inlet duct in the prototype. The working fluid in these tests was water having initial saturated, stagnation conditions ranging from 100 to 400 psia. All the tests were started from a nearly isothermal fluid condition. The water was stagnate before the decompressions were initiated.

Fig. I.1. Schematic of Blowdown Vessel.
ANL Neg. No. 900-3186.

internal consistency of the experimental measurements. The measured flow rate and thrust during the subcooled decompression are shown in Figs. I.3 and I.4, respectively. It is seen that the measured flow rate and thrust could be

Fig. I.2. System Configuration of Subcooled Calibration Test. ANL Neg. No. 900-4951.

One of the important parameters to be experimentally measured was the flow rate. This was determined by differentiating the mass decay signal of the weight transducer assembly which supported the entire blowdown vessel and its contents (see Fig. I.1). The resulting thrust was also measured with a similar load cell through a reducing lever arm assembly. The pressure inside the system was monitored at 15 locations, and two transient temperatures inside the internal skirt were measured.

The entire system was dynamically calibrated to ensure that the instrumentation and hardware would respond properly during the two phase decompressions. The calibration procedure was a subcooled blowdown, driven by an expanding gas volume as shown in Fig. I.2. In this manner, the state of the fluid at the upstream and exit locations of the exhaust duct was well known, providing a check on the

Fig. I.3. Comparison of Flow Rates in Subcooled Calibration Test and Calculated Flow Rates. ANL Neg. No. 900-5074 Rev. 1.

Fig. I.4

Comparison of Measured and Calculated Thrust in Subcooled Calibration Test. ANL Neg. No. 900-4996.

bracketed with an isentropic and isothermal expansion of the nitrogen gas within the internal skirt region (see Fig. I.2). It is also seen in Fig. I.3 that the flow rate calculated from the measured thrust with the relation

$$G = \left(\frac{F}{A_e v_l} \right)^{1/2} \quad (1)$$

where

G = mass flow rate per unit area,

F = thrust,

A_e = cross-sectional flow area at exit,

and

v_l = specific volume of saturated liquid,

and the flow rate calculated from the measured upstream stagnation pressure from

$$G = [2\rho_l(P_i - P_{atm})]^{1/2}, \quad (2)$$

where

ρ_l = fluid density of saturated liquid,

P_i = pressure at inlet to exhaust duct,

and

P_{atm} = pressure under atmospheric condition,

both fall within the envelope of the isothermal and isentropic expansions and also agree well with the flow rate determined from differentiating the measured mass-decay history. This indicated that the instrumentation in question and the hardware would respond properly during such a fast transient.

To characterize the two-phase critical flow rate and critical pressure ratio, a knowledge of both the upstream stagnation pressure and the upstream fluid quality is required. The upstream stagnation pressure was measured directly in these tests, but the upstream fluid quality was not. However, the upstream quality could be determined from the measured parameters. An overall momentum balance on the system permitted the fluid specific volume at the exit of the exhaust duct, v_e , to be determined from

$$v_e = \frac{1}{G_c^2} \left(\frac{F}{A_e} + P_{atm} - P_e \right), \quad (3)$$

where

G_c = critical mass flow rate per unit area,

A_e = cross-sectional exit flow area,

and

P_e = exit pressure.

The flow in the exhaust duct is assumed to be homogeneous, since the fluid momentum is due primarily to geometry induced acceleration. In addition, the fluid quality is assumed constant in the short duct length of three diameters, while the vapor phase is assumed to expand isentropically from the upstream to exit locations. Thus, the fluid quality, x , at the entrance to the exhaust duct can be expressed as

$$x = \frac{v_e - v_l P_{i0}}{v_{vi} \left(\frac{P_e}{P_{i0}} \right)^{-1/\gamma} - v_l P_{i0}}, \quad (4)$$

where

v_l = specific volume of saturated liquid,

P_{i0} = stagnation pressure at inlet to exhaust duct,

v_{vi} = specific volume of vapor at inlet,

and

γ = isotropic coefficient.

Combination of Eqs. 3 and 4 with the experimental measurements allow calculation of the upstream fluid-quality history shown in Fig. I.5. For comparison, Fig. I.5 also shows the isentropic upstream quality based on the measured upstream stagnation pressure. Back calculation of the total system energy inventory, using the inferred upstream quality and measured critical flow rate, agreed to within 1% of the actual value, thereby establishing confidence in this

calculation procedure. The departure of the inferred upstream fluid quality from the isentropic quality (see Fig. I.5) occurs at about the time when the downcomer annulus is voided of its initially contained liquid (see Fig. I.1).

Fig. I.5
Comparison of Inlet Fluid Quality
and Inlet Isentropic Quality. ANL
Neg. No. 900-4980 Rev. 1.

The continued rise of the inferred upstream fluid quality is apparently due to the vapor phase generated at the inside wall of the internal skirt and swept into the downcomer annulus, along with dispersed superheated liquid droplets from the interior of the internal skirt region. The combination of the downcomer geometry, little internal-surface area, and fluid resistance inside the internal skirt region promoted the preferential transport of the vapor phase, being formed on the inside of the internal skirt wall, up the downcomer annulus. The result was to indicate an upstream fluid quality greater than that inside the internal skirt region.

When the transient, two-phase critical flow rates from these large-duct tests are compared to the steady-state, small-duct, two-phase critical flow rates,¹ they correspond reasonably well, as indicated in Fig. I.6. The transient, large-duct critical flow rates appear to be slightly less than the steady-state, small-duct data. The small- and large-duct transient critical-pressure ratios (exit versus throat) are also found to agree, as seen in Fig. I.7. This indicates

Fig. I.6.

Comparison of Transient, Large-duct, Critical Flow Rates and Corresponding Steady-state, Small-duct, Critical Flow Rates of Starkman et al. ANL Neg. No. 900-5059 Rev. 1.

Fig. I.7

Comparison of Transient, Large-duct, Critical-exit Pressure Ratio and Corresponding Steady-state, Small-duct, Critical-throat Pressure Ratio Data of Starkman et al. ANL Neg. No. 900-5077 Rev. 1.

that the two-phase critical-flow phenomenon appears to be essentially the same in the transient case as in the steady-state case, and also nearly the

Fig. I.8. Comparison of Measured and Predicted Critical Flow Rates. ANL Neg. No. 900-5128 Rev. 2.

Fig. I.9. Comparison of Measured and Predicted Critical Pressure Ratios. ANL Neg. No. 900-5127.

same in the large-duct case as in the small-duct case. The implication is that the best presently available two-phase critical-flow analytical models that have been verified against the high-resolution, steady-state, small-duct, two-phase, critical-flow data can be applied to model the two-phase, critical-flow phenomenon in the transient, large-duct case.

When the critical-flow-rate predictions of the most widely used models^{2,3} are compared to the current transient data (see Fig. I.8), the Henry-Fauske model² is best. This is also true for the critical exit-pressure ratios, as seen in Fig. I.9.

Thermally dominated vapor growth is seen in Fig. I.10 to characterize the initial vapor growth. This volume may be inferred from measurements by

Fig. I.10. Comparison of Inferred Vapor Volume in Vessel and That Expected from Thermally and Inertially Dominated Bubble Growth. ANL Neg. No. 900-5516.

$$V_V = V_T - mv_\ell, \quad (5)$$

where

V_V = volume of saturated vapor,

V_T = total volume,

and

m = fluid mass.

Thermally dominated vapor-bubble growth in a superheated liquid may be given by

$$V_V = \frac{4}{3}\pi r^3 = \frac{4}{3}\pi \left(\frac{12}{11} \alpha_\ell Ja^2 t \right)^{3/2}, \quad (6)$$

where

r = bubble radius,

α_ℓ = thermal diffusivity of saturated liquid,

t = time,

and

Ja = Jakob Number

$$= c_\ell \rho_\ell (T_{\text{sat}} - T) / \rho_v h_{v\ell},$$

where

c_ℓ = specific heat of saturated liquid,

T_{sat} = temperature for saturated condition,

T = temperature,

ρ_v = fluid density of saturated vapor,

and

$h_{v\ell}$ = latent heat of vaporization.

Inertially dominated vapor-volume growth given by

$$V_V = \frac{4}{3}\pi r^3 = \frac{4}{3}\pi \left(\frac{P_{\text{sat}} - P}{\rho_\ell} \right)^{3/2} t^3, \quad (7)$$

where

P_{sat} = pressure under saturated condition

and

P = pressure,

is also shown in Fig. I.10 for comparison, but it does not correspond to the inferred vapor volume after the first 10 ns.

An analytical model based on bubble-growth theory has been developed that predicts the measured internal vessel pressure histories in these decompressions quite well. The resulting predicted internal-vessel-pressure histories are dependent upon the amount of internal surface area and upon the ratio of exit flow area to internal flow area. Such relationships appear to be intuitively reasonable.

C. Transient Critical Heat Flux (J. C. M. Leung and O. C. Jones, RAS)

Current analysis of water-reactor-system behavior following a loss-of-coolant accident (LOCA) assumes that critical heat flux (CHF) in the transient system can be determined by steady-state correlations. Consequently, when the core flow reverses following an inlet-coolant-pipe rupture, the assumed CHF becomes zero and instantaneous dryout is postulated. Various experiments have demonstrated that such instantaneous dryout is not achieved for fast transient systems, and large integral-test programs are presently underway to determine the actual time to dryout for rod bundles in various systems. In order to interpret the experimental results from these large-scale, integral-test programs, fundamental experiments on the basic mechanisms are required. Such an experiment is in progress at ANL.

The experiment uses Freon-11 as the boiling fluid in a transparent annular test section, so that direct photographic observations can be made of the fluid behavior before, during, and after local dryout. The experimental apparatus (shown in Fig. I.11) basically involves a supply vessel, which is supported by a weight transducer, a transparent test section, a variable volume blowdown vessel, which is also supported by a weight transducer, and a large, low-pressure receiving vessel.

Before initiation of the flow transient, steady-state conditions are developed by flowing the fluid from the supply vessel vertically upward through the test section, and into the receiving vessel. The flow transient is initiated by valves that isolate the supply vessel from the test section, close the path from the top of the test section to the receiving vessel, and open a path from the bottom of the test section to the receiving vessel. The solenoid valves are actuated simultaneously.

After initiation of the transient, the system decompression is determined by the thermodynamic conditions of the system during steady-state operation and the volume available in the variable-volume blowdown vessel. The volume of this vessel is changed for various runs in order to investigate different system depressurization rates.

Fig. I.11. Transient-Blowdown Critical-heat-flux
Test Loop. ANL Neg. No. 900-4557.

Measurements recorded during the transient are (1) test-section mass flow rate, determined by differentiating the weight versus time response in the variable-volume blowdown vessel, (2) test-section pressures, (3) temperatures, and (4) inlet and outlet void fractions as determined by turbine meters and also capacitance void probes. As mentioned above, the transparent outer wall of the test section will permit direct photographic observation of the fluid during decompression.

The results of this experimental investigation will illustrate the relationship between the transient critical heat flux, the system depressurization rate, local void fractions, and two-phase flow regimes. Such information is not only essential for interpreting large-scale, integral-test results, but it can also play a major role in determining what instrumentation should be used for a given test program, and also where such instrumentation should be located.

Construction of the experimental apparatus and calibration of various instrumentation have been completed. Heat-balance and heat-loss tests have been performed on the apparatus, and the results are satisfactory. The experiments are planned to be carried out at two levels:

1. Steady-state CHF tests at different (a) pressure up to ~70 psia, (b) mass velocity, and (c) inlet subcooling.
2. Blowdown characteristics and transient CHF tests.

Adiabatic blowdown tests and some steady-state CHF runs have already been performed.

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II. TRANSIENT FUEL RESPONSE AND FISSION-PRODUCT RELEASE PROGRAM

Responsible Group Leaders:

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The objective of this program is to develop a physically realistic model describing the release of fission products from irradiated Light-Water Reactor (LWR) fuel as a function of thermal transients anticipated in hypothetical accident situations. Such accidents might be generated by power-coolant mismatch (PCM), Boiling-Water-Reactor (BWR) flow blockage, loss-of-coolant-accident (LOCA), or reactivity-initiated-accident (RIA) conditions that can result in a wide range of heating rates and temperature profiles in the fuel. Out-of-reactor experiments to simulate and study in-reactor fuel and fission-product phenomena will be made, and a computer model of fuel-fission-product interactions that can be used with the Fuel-Rod Analysis Program (FRAP) code system being developed by the Aerojet Nuclear Company (ANC) for LWR safety analyses will be developed.¹ The program parallels and uses the experience of a similar Argonne National Laboratory (ANL) safety-oriented program (189a Budget Activity No. 02-647, "Fuel Response to Applied Transients") in support of the development of Liquid-Metal Fast-Breeder-Reactor (LMFBR) fuels.

A Program Description² has been prepared, which was approved by the Fuel Behavior Branch of the Reactor Safety Research Division (RSR). The following overall approach shows the interrelationship of the experimental and analytical efforts in this program:

1. Fission-gas Behavior and Fuel Response

a. Develop a preliminary steady-state and transient model for predicting LWR fuel response and fission-gas-release behavior based on the existing LIFE/GRASS code system.

b. Verify the model and contribute to baseline data in an experimental program on irradiated and unirradiated UO₂ fuel by (1) characterizing LWR-irradiated fuel; (2) simulating LWR-accident heating conditions in out-of-reactor direct-electrical-heating (DEH) experiments and analyzing the released fission products and characterizing the fuel; and (3) characterizing fuel from ongoing, related RSR test programs in the Power Burst Facility (PBF) and in the Halden Reactor.

c. Modify the analytical treatment and develop improved correlations.

d. Iterate with the experimental program for additional improvement of the model as necessary.

e. Assist ANC in the integration of the model into their FRAP Transient (FRAP-T) accident-analysis code system.

2. Volatile Fission-product Behavior

a. Derive basic input data from the experimental program described above.

b. Develop models for incorporation into the GRASS/FRAP-T code system.

c. Verify the models experimentally and iterate as necessary.

d. Assist ANC in the integration of the model into FRAP-T.

A. Modeling of Fuel-Fission-Product Behavior (J. Rest, MSD)

The analytical effort will provide a link between our simulated transient-heating test program and the analysis of LWR accidents in the Thermal Fuel Behavior Program of ANC (TFB-ANC) to ensure that the experimental program is relevant to the accident conditions of interest and the results of the experimental program are available in a useful form. The initial objective is to develop a fission-gas-release and fuel-swelling model for irradiated LWR UO_2 fuel based on similar work at ANL on $(\text{U}, \text{Pu})\text{O}_2$ fuels for Fast-Breeder Reactors (FBR). The emphasis of the analytical program will then shift to the volatile fission-product metals and oxides.

The inert fission-product gases, xenon and krypton, are basically insoluble in the fuel matrix and are known to precipitate into gas bubbles and cause fuel swelling. The gas atoms or bubbles may diffuse or migrate to surfaces in the fuel, resulting in release of gas. The extent of fuel swelling and gas release during the accident depends upon the type and duration of the accident, the temperature distribution, the prior burnup, the fuel microstructure, and the state of the retained fission gas. A computer-based model (GRASS) has been developed and used for the calculation of steady-state fission-gas behavior in fast-reactor fuels.³ This model includes the effects of burnup, attraction to and release from structural defects, diffusion, migration, bubble coalescence, re-resolution, temperature, and temperature gradients on the distribution of fission gas within the fuel and on the amount of fission gas released from the fuel. In this program, the GRASS model, with steady-state and transient capabilities,⁴ is being used as a basis for analysis of the behavior of fission gases in LWR fuels during accident-heating conditions. The validity of the analysis will be determined by means of our experimental program.

An LWR version of the GRASS code has been developed by modifying the existing LMFBR GRASS code. This preliminary LWR GRASS code has been

generalized to include a multiregion calculation (up to 10 axial sections and 20 radial rings) and a restart capability. The code's efficiency has also been increased; the steady-state LWR GRASS code run is 40% faster than its LMFBR predecessor. A preliminary version of the transient LWR GRASS code is now complete, has been communicated to ANC for inclusion into FRAP-T,⁵ and has been described at the NRC Modeling Review Meeting on March 12-13, 1975. A description of the LWR-irradiated fuel microstructure has been included in GRASS. The evolution in time of the fuel microstructure and the fission-gas-bubble size distributions is now being calculated for a variety of fuel types and steady-state operating conditions using input data which were supplied to ANL by ANC, for Westinghouse CVTR and Saxton test rods. Code calculations will be compared with measured gas-release data from these representative LWR-irradiated fuels, which will serve as a check of the steady-state analysis. Other checks for the code calculations will include the existing correlation developed by Beyer and Hann⁶ and the results of ANC's study in the Halden Reactor of the influence of prior power history on fission-gas release.⁷ The steady-state analysis will provide the necessary initial conditions to the transient GRASS code at the start of the LWR accident.

At present, LIFE-III⁸ is being used as a driver code for the GRASS subroutine; FRAP-S (FRAP-Steady State), being developed by ANC, is not yet at a usable stage. LIFE-III has been modified for LWR use by including the conversion of relevant fuel and cladding properties and behavior from FBR-irradiated mixed-oxide to LWR-irradiated UO₂ fuel and by taking into account the radial flux depression in temperature calculations (supplied by ANC). An on-line graphics capability has been developed for comparison of analytical and experimental results, which will facilitate the analysis of fuel behavior.

B. Experimental Technique

1. Direct-electrical-heating Apparatus (B. J. Wrona, J. A. Buzzell, and D. R. Pepalis, MSD)

Direct-electrical heating was described by Freund and Schikarski⁹ as a method of heating oxide fuels through the melting transition; the technique has been demonstrated successfully in several laboratories.⁹⁻¹¹ Recent work at ANL has shown that LMFBR-irradiated mixed-oxide fuel pellets can be heated to fuel ejection and collapse using this technique.¹² The design of the DEH apparatus developed for the in-cell accident-simulation experiments on LMFBR fuels was used as the basis for the simulation apparatus for this LWR program; modifications were necessary to the electrical power supply and the design of the specimen chamber to satisfy the more sophisticated requirements of the LWR-fuel-transient test program and to improve remote operation. The LWR-DEH apparatus, along with associated apparatus for collecting fission products, is shown schematically in Fig. II.1. Proof-testing of the new LWR-DEH chamber with unirradiated UO₂ pellets is shown in Fig. II.2.

Fig. II.1. Schematic Design of In-cell DEH and Fission-product Collection Apparatus. Neg. No. MSD-183709.

Fig. II.2

DEH Apparatus Specimen Chamber Being Proof-tested Out of Cell, Using Unirradiated UO₂ Pellets. Neg. No. MSD-183586.

As with the LMFBR-DEH apparatus,¹³ the thermal-simulation apparatus for this program was design to provide direct visual observation of fuel-fission-gas phenomena during transients. Electrical heating of the fuel stack is automatic and continuous through the use of a two-mode electrical circuit. The apparatus is capable of operating in both steady-state and transient-heating modes, and up to 1500°C/s heating rates are within the capabilities of the electronics.

Improvements have been made over earlier designs to facilitate remote stacking of the pellets by means of hot-cell manipulators. The DEH chamber is hinged at one corner to permit loading a fuel pellet or stack of pellets between electrodes in the horizontal position and then rotating to the vertical position for operation. To allow for axial displacement of the fuel stack during temperature-cycling operations, the upper electrode is bellows loaded. Tungsten electrodes with end diameters equivalent to the pellet diameter are used to ensure uniform loading of the pellet stack. Also, provisions have been made for varying the load on the fuel column.

During the test, the test section is cooled continuously by flowing helium gas, and changes in specimen surface temperature can be achieved by modifying the flow rate. Early problems with vapor fogging of the observation window have been resolved by sweeping the window with a stream of helium.

Fig. II.3. Diagram of Electrical Circuit for DEH. Neg. No. MSD-60663.

The electrical power supply (Fig. II.3) consists of a low-voltage, high-current power supply (100 V and 100 A) and a high-voltage, low-current power supply (2500 V and 4 A) operating in parallel, with their positive terminals decoupled by diodes. This arrangement is more efficient than a single power source capability of 2500 V and 100 A. Current can be drawn only from the low-voltage power supply when the voltage drop across the stack is <100 V. When the resistance of the UO_2 pellet stack is such that the potential drop across the pellet stack exceeds 100 V, the diodes back-bias and shut off the low-voltage power supply. At start-up, the high-voltage power supply serves to initiate current flow in the high-resistance pellet stack in lieu of an external heating device.

Voltage- and current-limiting features are used to engage and assume control automatically when either the voltage or current preset control levels are reached. Experience has shown that, after the pellet-stack temperature

has increased to a high level, power control can lead to a runaway condition of high current. The current-limiting feature takes over when a preset current is reached, and the mode of operation of the power supply becomes current regulated rather than power regulated. A second current limiter was added to the electrical system, in parallel with the first, which is used to initiate melt-down of the pellet stack. Thus, the previously detrimental condition of runaway current has been utilized to initiate the transient phase of the experiment.

A timer has been installed to interrupt the simulated transient at various stages before the actual onset of gross fuel motion and to permit the total time of the test to be reproduced accurately.

A power-ramp rate switch has been installed to permit power ramps to be achieved at rates that can be varied as desired. Thus the energy-input rate that causes gross fuel motion can be reproduced within small experimental error, and the timer allows termination of subsequent experiments at various time intervals before fuel slumping occurs. This permits the study of fission-gas and volatile fission-product behavior during progressive stages of the simulated accident.

Slumping of the fuel (i.e., stack-length reduction) is monitored by a Linear Variable Differential Transformer (LVDT) connected to a high-speed recorder. In addition to the amount of expansion during heating and contraction during slumping, the LVDT also gives a numerical value to the rate at which slumping occurs because of its rapid response time. The response of the LVDT has been excellent in all tests to date.

Following gross fuel motion in which fuel slumping exceeds the ~0.130-in. travel of the upper electrode with its attached LVDT, arcing usually occurs across the gap between the pellet stack and the upper electrode, unless the input power is instantaneously shut off. This arcing can lead to larger melt fractions than were actually achieved during the test. To eliminate the necessity for rapid human response, provision has been made for automatic termination of the input power.

The bellows attached to the upper electrode causes resistance to motion, which can be avoided if the lengths of the pellet stack and the tungsten electrodes are optimized; variation in pellet-stack length requires simultaneous alteration of the electrode length so that the bellows and the upper electrode can follow fuel expansion during heating and gross fuel motion on slumping.

Acoustic-emission transducers, which permit the detection of lower intensity sounds that occur during thermal-shock cracking, are being developed for use with the DEH apparatus. Preliminary tests indicate this technique will permit insight into the role of, for example, cracking during the simulation of accidents or cracking during thermal-cycling experiments below the failure threshold.

Fuel motion during rapid transients is recorded on 16-mm color movie film using a high-speed movie camera. The camera speed can be varied from 24 to 12,000 frames/s, but generally a speed of 1000-2000 frames/s is used. The high-speed camera has a short lag time before it attains a preset speed level; therefore, the camera is started before the transient is initiated. To synchronize the start of the transient on both the recorder and movie, the switch (current limiter No. 2), which initiates the transient, is wired so that a 1/1000-s flash is set off by a flash attachment at the start of the accident.

Auxiliary heating elements are being developed for externally heating the surface of the test fuel column to simulate radial temperature gradients anticipated in hypothetical LWR-accident situations. Methods for simulating thermal conditions in which the fuel-centerline temperature decreases while the fuel-surface temperature increases (LOCA and BWR flow blockage) are also being investigated.

2. Correlation between Nuclear and Electrical Heating (J. C. Vogelwede, MSD)

The correlation between nuclear-accident-heating and electrical heating conditions has been established on the basis of representative fuel-temperature predictions calculated by ANC for their LWR-simulated accident tests to be conducted in the PBF.^{14,15} Fuel-stack temperatures during electrical heating were calculated as a function of radial position and time using the Direct Electrical Heating Transient Temperature Distribution (DEHTTD) Code. This program normally accepts time-dependent power- and surface-temperature histories of the DEH fuel stack to calculate internal temperature distributions. The reverse of this process was used to correlate electrical- and nuclear-accident-heating conditions, that is, typical DEH power- and surface-temperature histories were generated by following the ANC representative fuel-temperature profiles. Several points should be noted on the basis of these correlations:

a. To follow each of the ANC representative fuel-temperature profiles, a history of DEH power- and surface-temperature values must be followed. It is not known to what degree the DEH surface-temperature values may be controlled during transient tests. The mean power dissipation characteristics of a fuel stack are determined by experimental configurations (e.g., clad or unclad fuel, whether heat shields are used, etc.). Once the power input and the experimental configuration have been established, the surface temperature may be varied a few hundred degrees centigrade from the mean value by adjusting the rate of helium-coolant flow.

An analysis of DEH power-dissipation characteristics for different experimental configurations has been initiated. This will allow a better prediction of DEH transient behavior before tests are conducted.

b. Analysis of ANC-calculated fuel-temperature profiles indicates a wide range in surface power dissipation, as can be seen in the selected curves in Fig. II.4. Temperature profiles during a PCM accident prior to critical

(a) PCM--Power-cooling mismatch accident prior to occurrence of critical heat flux with a peak-power ramp rate of 6 kW/ft/min¹⁴

(b) BWR flow blockage accident: 100% area blockage¹⁴

(c) LOCA--Loss-of-coolant accident: Cold-leg breakthrough blowdown assuming that heat transfer is entirely by natural convection to steam and allowing metal-water reaction¹⁴

(d) RIA--Reactivity Initiated Accident: Transient power and the resulting BWR-6 fuel rod temperatures for a 8×10^5 J/kg (200 cal/g) reactivity initiated accident from hot standby reactor conditions¹⁵

Fig. II.4. Selected ANG-calculated Fuel-temperature History Curves

heat flux (Fig. II.4a) show surface power dissipation of $\sim 100 \text{ W/cm}^2$ at the initiation of the accident and $\sim 300 \text{ W/cm}^2$ 150 s later. DEH response of this magnitude has been demonstrated repeatedly.

A reverse behavior is shown by the fuel-temperature profiles during LOCA and BWR flow-blockage accidents (Figs. II.4b and II.4c). The plot for the BWR flow-blockage accident indicates that the initial surface power dissipation is again $\sim 100 \text{ W/cm}^2$. By the end of the accident, this value is virtually nil because of the coolant blockage. In the DEH environment, this means power dissipation from the fuel stack must be eliminated in the space of a few seconds. Current studies indicate this may be approximated through the use of external heating elements.

c. The present heating capability of the DEH ($\leq 1500^\circ\text{C/s}$) may be too low to simulate the very rapid initial heating ramp of the RIA. This heating limitation is based on existing DEH power supplies; an adequate source of power should permit modeling even the initial ramp of the more severe RIA (Fig. II.4d).

The temperature gradient in the electrically heated fuel stack is computed using a concentric-shell approximation and recorded measurements of fuel-surface temperature and electrical power or current.^{3,8} Steady-state and transient-heating computer programs have been completed.⁸ Preliminary use of these programs has identified several problem areas that are presently the subject of intensive study. In addition, operation of the DEH apparatus has revealed problems in surface-temperature measurement that require early solution. The major areas of concern are summarized below.

The temperature dependence of electrical conductivity in fuel materials results in a large variation in the power density of DEH test specimens. Centerline power-generation rates will be greater than those at the surface. To simulate reactor conditions, where centerline power density may be a factor of two lower than the surface, power input and the surface temperature of the DEH apparatus must be varied to approximate nuclear conditions. This matching capability has been developed, and DEH/nuclear matching techniques are being developed for each of the ANC-accident types. These methods are limited at high temperatures by the sparsity of property data for fuels.

Fuel-surface temperatures are measured continuously using an Ircon infrared pyrometer, which has a response time of the order of 0.1 s. Accurate surface-temperature measurement is influenced by (a) fuel emissivity, (b) fuel fission-product vapors, and (c) the intervening specimen chamber and hot-cell windows. The latter can be taken into account by calibration, but vapors are not uniformly produced, and, unless removed immediately, they significantly reduce recorded fuel temperatures. Finally, a wide range exists in reported emissivity values for UO_2 , which can result in fuel-surface temperature measurement errors of as much as $\pm 200^\circ\text{C}$. Work is in progress to resolve the

emissivity problem, and fuel-vapor deposition on the windows has been eliminated by the use of a directed flow of helium.

3. Fission-product Collection System (W. D. Jackson and L. R. Kelman, MSD)

The fission-product collection system, shown schematically in Fig. II.1, has been designed with charcoal traps to permit quantitative determination of the amount of released fission gases and tantalum traps to permit qualitative determination of released condensible solid fission products. The system is designed for minimum back pressure in the DEH chamber. Components of the system are being procured and fabricated.

A large fraction of the emitted volatile fission products and all fission gases will be carried by flowing helium coolant through a coarse prefilter to remove large particles. The volatile fission products condense on tantalum plates inside the chamber and on tantalum Raschig rings in a series of 6-in.-long, $1\frac{1}{2}$ -in.-dia stainless steel capsules, which start from a position $\sim 1\frac{1}{2}$ in. from the fuel column. The capsules are flanged together in a manner that will permit remote disconnection for separate chemical analysis. Tantalum was chosen because of its resistance to attack by the chemicals that will be used to dissolve the fission products for analysis. External cooling of the capsules is provided to encourage condensation of volatiles. Sufficient capsules will be arranged in series to ensure the removal of all condensibles from the gas stream. Thermocouples are provided to monitor the temperature along the length of the trap and to permit determination of the composition of the condensibles as a function of deposition temperature.

Sufficient in-cell piping follows the tantalum trap to allow extraction of heat from the gas stream, which will prevent damage to the Millipore final filter that removes the remaining fine particles. Cooling of the gas stream also prevents excessive use of liquid nitrogen in the fission-gas trap.

A single 293-mm-dia Millipore filter has been chosen to prevent particles from entering the fission-gas trap. Two filter media will be available for testing; a low-temperature, acid-soluble media of 0.45- μ m pore size is the most desirable from an efficiency and analytical viewpoint, but a higher temperature, acid-leachable media with 5.0- μ m pore size will be available if needed.

A 73-liter, 12.5-in. by 36-in. dewar has been selected to provide a liquid-nitrogen cryogenic environment for the precooler and two charcoal traps. The precooler coil is ~ 35 ft of $1\frac{1}{2}$ -in. tubing. The charcoal traps are $1\frac{1}{2}$ in. in diameter by 24 in. in length and are in series. Although one trap of this size is theoretically adequate, two traps are used for added assurance. A small additional trap, shown in Fig. II.1, can be added, if deemed necessary, to monitor the efficiency of the first traps or to permit bypassing. The completely stripped sweep gas will be dumped into the cave exhaust system.

4. Hot Cell (W. D. Jackson and L. R. Kelman, MSD)

A safety review and a preliminary safety analysis have been performed of the DEH, fission-product collection system, and fuel-recovery operation in an existing hot cell not previously used for fuel work. The basis for the analysis was a maximum credible accident that assumed two contingencies: (a) the sweep-gas control system malfunctions, which overpressurizes the experimental apparatus, and (b) the weakest link of the containment system, the observation-photography window, bursts. One unlikely event, clogging of the cell exhaust system, could lead to cell pressurization and leakage into the work area. The safety analysis was favorable, provided several cell and equipment modifications are made to assure compliance with all relevant safety regulations.

Cell modifications necessary to improved safety, efficiency, and convenience in operation include a sampling system on the cell air exhaust; isolation of the LWR-DEH cell from adjacent cells by additional shielding and by sealing intercell transfer doors; sealing the cell from external work areas by booting manipulators, gasketing access plugs, and upgrading the seal of the contamination transfer door; and containing the fuel-recovery apparatus to minimize spread of active debris. These modifications are being implemented.

5. Extraction of Irradiated Fuel from Cladding (L. R. Kelman, B. J. Wrona, F. E. Savoie, and J. A. Buzzell, MSD)

To perform DEH experiments on irradiated fuels, an integral stack of fuel must first be removed from the cladding so that the cladding will not short circuit the electrical current. Fuel removal is complicated by the extensive cracking normally found in irradiated oxide fuels. Mixed-oxide fuel irradiated in EBR-II to 3-5 at. % burnup was successfully "extruded" from its stainless steel cladding, using a device that has been described in detail in Ref. 12. Experiments have been run which verified that the low expansion coefficient of Zircaloy precludes the use of this method on Zircaloy-clad LWR fuel.

Attempts were made to remove an integral stack of fuel by longitudinally slitting through the cladding of the high-burnup experimental BWR-irradiated fuel element described in Sec. II.C.3 below. The first attempt, using a ceramic cutoff wheel in a makeshift setup, was unsuccessful. The type and extent of cracking of the original fuel is shown in Fig. II.5b. The fragmentation of the fuel that resulted from the first attempt at recovery can be seen in Fig. II.5a. The large fuel particles were extremely hard; they did not break into smaller particles even when encouraged to do so. Metallographic examination of a cross section of the fuel element showed metallurgical bonding in some areas of the fuel-cladding interface, Fig. II.5c. Subsequently, a stack of fuel was recovered by careful slitting of the cladding in a much more rigid and controllable setup. First, $\sim 120^\circ$ of cladding was removed; the fuel

was impregnated with paraffin to bond the particles together; and, finally, a heat-shrinkable plastic tubing was used as insurance. Although this approach can be used to recover some fuel specimens, it appears to be more time consuming than other promising methods. From this experience, however, it is evident that paraffin can be used to bond the particles of fuel together to keep the pellets intact while being handled at low temperatures by any of several methods we are now exploring for extracting an intact column of fuel.

(a) Fuel Pieces Resulting from First Attempt at Fuel Recovery. Mag. 1.4X. Neg. No. MSD-183072.

(b) As-polished Cross Section of Fuel Element Showing Coarse Cracking. As-polished. Mag. ~213X. Neg. No. MSD-182887.

Cladding

Fuel

(c) Metallurgical Bonding of UO_2 Fuel to Zircaloy Cladding. As-polished. Mag. 250X. Neg. No. MSD-182894.

(d) Intact Fuel with ~120 Degrees of Cladding Removed by Slitting. Mag. 1.75X. Neg. No. MSD-183073.

Fig. II.5. Structure of BWR-irradiated GE-SA-1 No. B58 Fuel Element and Results of Early Attempts to Extract an Intact Column of Fuel for DEH Testing

Two particularly promising potential fuel-removal methods are now being explored. In one approach, the Zircaloy cladding is heated rapidly by electrical resistance, and, before the fuel has had a chance to heat appreciably, it is pushed out of the cladding. In the other, the clad fuel is supported in the DEH chamber, the cladding is heated with a heat gun or other device for rapid heating, and the upper DEH electrode is used to push the fuel out of the cladding. The fuel used initially in these studies was unirradiated UO_2 pellets that were thermally shocked to simulate the cracking of irradiated fuel. The cracked pellets were then clad in undersized Zircaloy tubing by shrink fitting.

C. Experimental Program

1. Experimental Plan (L. R. Kelman, L. A. Neimark, and D. Stahl, MSD)

The experimental program will provide a basis for verification of the steady-state and transient LIFE/GRASS analyses as well as the basic input data for development of the fission-product behavior module. The experimental program, which was discussed in detail in the Program Description,² will consist of three basic parts: (a) characterization of LWR-irradiated fuel elements, (b) characterization of PBF- and Halden-tested fuel elements, and (c) DEH experiments.

Characterization of the total fission-product behavior of representative LWR-irradiated fuel elements will provide code verification for LIFE/GRASS steady-state analyses and also the baseline data from which to assess the effect of accident situations on fission-product behavior. Characterization of PBF- and Halden-tested fuel elements will allow comparison with the DEH test results and the GRASS code analyses and better establish the credibility of the analyses for the actual LWR-accident conditions. A limited amount of PBF- and Halden-tested fuel will be characterized to verify the validity of the DEH input to the GRASS code.

The DEH experiments on unirradiated and irradiated fuel will be used to follow the entire scenario of fuel behavior from steady state through the accident-heating conditions to provide verification of the steady-state and transient codes. The steady-state experiments will be performed to obtain kinetics data on fission-product release in irradiated fuel. The transient experiments will be used to provide direct verification of the GRASS analyses for selected accident situations and to generate information on volatile fission-product behavior that will be included in the fission-product behavior module. Fuel, from tests terminated at progressive stages of the simulated accident, will be characterized for fission gas and solid fission-product behavior. The released fission products (iodine, cesium, tellurium, and volatile oxides, as well as the fission gases) will be identified by qualitative analysis of the collected effluent. A quantitative determination of the fraction of fission gas released will be made by posttest fuel analysis and comparison with sibling fuel.

The preliminary test plan for the transient experiments in the DEH will be based on the heating conditions established by ANC for LWR-simulated accident tests to be conducted in the PBF.^{1,15} Some selected fuel-temperature history curves calculated by ANC for several types of LWR accidents (a) PCM, (b) BWR flow blockage, (c) LOCA, and (d) RIA were reproduced in Fig. II.4. The accident types and the heating conditions derived from the ANC curves are given in Table II.1, which presents a minimum test matrix for the desired conditions. The final test matrix will depend upon a finer delineation of the accident conditions; it is conceivable that a single test may yield information for more than one accident type. Conversely, it may be necessary to expand the matrix if code verification/development requires a broader test spectrum.

TABLE II.1. Preliminary DEH Transient Test Plan

Accident Type	Experimental Parameters				Minimum No. of Tests					
	Heating Rate, °C/s		Fuel Burnup ^a	Restraint	Unrestrained ^a			Restrained ^a		
					U	M	H	U	M	H
PCM	2-10	200-300	M,H	No/Yes	-	3	3	-	1	1
Flow Blockage	60-85		M,H	No	-	3	3	-	-	-
LOCA	150-300 ^b		U,M,H	No/Yes	1	3	3	1	1	1
RIA	1000-1500		U,M,H	No/Yes	-	1	1	1	2	2
Total Tests ^c					31					

^aU - unirradiated, M - medium burnup, and H - high burnup.

^bNRC-RES has suggested lower heating rates for the fuel surface during a LOCA -- e.g., 50°C/s during blowdown and even slower transients during refill and reflood.

^cApproximately 15 additional tests are anticipated to verify (1) the reproducibility of DEH test results, (2) the steady-state version of GRASS, and (3) the code analysis for the effects of prior power level.

The preliminary DEH transient test plan in Table II.1 calls for a minimum of 31 tests -- 28 tests with irradiated fuel and three tests with unirradiated fuel. As many as 15 additional tests may be required to verify the reproducibility of the DEH test results, provide supplemental information for code verification and development, and verify the code analysis for the effects of prior power level.

2. Experimental Results (B. J. Wrona and T. M. Galvin, MSD)

DEH tests to date have been limited to two areas: preliminary out-of-cell scoping studies, in conjunction with the development of the LWR-DEH equipment and methods; and the first studies of the mechanical response of

unirradiated UO_2 during simulated accidents. Depleted, unirradiated, unclad UO_2 pellets (0.342 in. dia by 1.14 in. long), weighing ~17.7 g, were used in these preliminary tests.

In general, the DEH chamber and associated instrumentation have functioned well during the out-of-cell proof tests (Fig. II.1). The electrical power supply and controls of a prototype DEH system were used in these studies because the power supply and controls for the LWR-DEH apparatus were not completed. A current of ~145 A was required to melt the LWR-diameter UO_2 pellets. This current is beyond the capacity of the present borrowed transformer; a new, larger transformer has been ordered.

In the mechanical-response studies, direct observations of fuel motion during simulated accident tests in the DEH, together with pretest and posttest metallographic characterization, will permit quantitative evaluation of fuel-failure mechanics.

Figure II.6a is a macrophotograph of two as-sintered UO_2 pellets that were DEH tested at a power level below the threshold of gross fuel motion. Some molten fuel can be seen in the large cracks. Figure II.6b shows the lower pellet from a similar test run at a considerably higher power level. Most of the upper pellet melted and flowed through the large crack in the lower pellet. As seen by a comparison of Fig. II.6a with Fig. II.6d, the melt fraction and structure of these DEH-tested 0.342-in.-dia UO_2 pellets are similar to those of some 0.2-in.-dia mixed-oxide pellets that were transient over-power tested in the TREAT reactor. Evidence from these tests indicates that the width of cracks in the fuel considerably influences the type and extent of motion of molten fuel during simulated accidents.

3. Fuel Specimens (L. R. Kelman, MSD)

Irradiated UO_2 fuel, identified for the integral in-reactor experiments of the TFB-ANC program, will be used in this program. For the near term, to satisfy the need for the development of a fuel-extraction method and to conduct preliminary DEH checkout and scoping tests, two ~40-in.-long fuel rods were obtained late in October 1974 from ANC--a low-power (4-5 kW/ft), low-burnup (6600 MWd/MTM average) rod, MAPI No. M12 irradiated in Saxton, a PWR;¹⁶ and a medium power (<14 kW/ft), high-burnup (31,000 MWd/MTM average) rod, GE-SA-1 No. B58 irradiated in the Vallecitos and Dresden-I reactors, both BWR's.¹⁷

To conduct the comprehensive program that begins with the proposed DEH tests, NRC-RES has arranged to obtain irradiated fuel from a commercial power reactor, Carolina Power and Light Company's Robinson Reactor, Unit No. 2. Medium- and high-burnup fuel will be used in the first series of tests. An estimated average of two fuel pellets will be used in each test, with an expected range of one to three. Based on a 50% success in decladding pellets

(a) DEH Test at Power Level below the Threshold of Gross Fuel Motion Showing Molten Fuel Solidified in the Large Cracks. Mag. $\sim 2.25X$. Neg. No. MSD-60958.

(b) DEH Test at High Power Showing that Melted Fuel from the Top Pellet has Exuded through a Crack in the Bottom Pellet. Mag. $\sim 2.5X$. Neg. No. MSD-60956.

(c) Radial Melt Fraction at Axial Midplane of Pellets Tested below the Threshold of Gross Fuel Motion. Mag. $\sim 7X$. Neg. No. MSD-60957.

(d) Similar Radial Melt Fractions in TREAT-tested Pellets. Mag. $\sim 11X$. Neg. No. MSD-163588.

Fig. II.6. Preliminary DEH Mechanical-response Tests of Bare As-sintered 0.342-in.-dia UO_2 Pellets (a, b, and c) and Comparison with Clad 0.2-in.-dia Mixed-oxide Pellet That Had Been Transient Tested in TREAT

and a DEH test success of one in three, ~500 irradiated fuel pellets will be needed for the preliminary DEH transient and steady-state test program.

About 50 unirradiated fuel pellets will be required to satisfy the preliminary DEH test program and to provide baseline and supportive information on fuel behavior. Archive unirradiated pellets from the same lot as the Robinson fuel are being sought, with the help of ANC, from Carolina Power and Light and Westinghouse. So far these efforts have been unsuccessful. Archive fuel from ERDA's LWR experimental program is a possible backup source of unirradiated fuel pellets for our program.

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III. MECHANICAL PROPERTIES OF ZIRCALLOY CONTAINING OXYGEN

H. M. Chung, A. M. Garde, and T. F. Kassner, MSD

The objective of this program is to obtain a better understanding of the effect of oxidation on the mechanical behavior of Zircaloy to evaluate the acceptance criteria for Emergency Core Cooling Systems in light-water reactors. At present, the criteria stipulate a maximum cladding temperature of 1200°C and a maximum total oxidation of 17%, which is defined as the amount of wall thickness converted to oxide when all oxygen present in the Zircaloy is assumed to be in the oxide phase. These criteria implicitly assume that the metallic β -phase in Zircaloy, which exists at temperatures above ~860°C, is not sufficiently contaminated with oxygen at high temperatures to be brittle during a loss-of-coolant-accident (LOCA) reflood.

The strength and ductility of Zircaloy-2 and -4 will be determined as a function of temperature, oxygen content, microstructure, and strain rate by performing uniaxial tensile, four-point-bend, biaxial tube-burst, and impact tests. Internal expanding-mandrel loading may also be employed primarily for fuel cladding containing high oxygen concentrations (>1 wt %). The mechanical test conditions will cover the temperature range from ambient to 1400°C, strain rates between 10^{-4} and 10^{-1} reciprocal seconds, internal pressures in the range of from 250 to 3000 psi at the test temperature, and several cooling rates from 1400°C to the lower test temperatures. Although this range of test variables encompasses the conditions of interest in postulated LOCA situations, the mechanical-property results will also provide a data base for fuel-element modeling codes used to predict cladding deformation for power-coolant mismatch (PCM) situations.

A. Experimental Methods and Test Matrix

1. Specimen Materials

A specification was written to procure the Zircaloy-2 and -4 sheet stock for fabrication of the uniaxial test specimens. The compositions of the materials obtained from AMAX Specialty Metals and Teledyne Wah Chang are given in Table III.1 along with the grain size, thickness, and heat treatment. A preliminary X-ray diffractometer scan of the Zircaloy-2 sheet indicated the expected high (001) intensity in the plane of the rolled sheet.

2. Specimen Geometry

Uniaxial specimens with a 1-in. gauge length and a 1/4-in. width, fabricated according to ASTM specification E8-69, are being used in the first part of this investigation. In these specimens the gauge length is parallel to the rolling direction. Transverse specimens will be used in the future, depending on the results obtained with the longitudinal specimens. Each shoulder region is reinforced on both sides by spot welding supporting coupons of the same material to the grip area.

TABLE III.1. Source and Composition of the Zircaloy-2 and -4 Materials Used in the Fabrication of the Uniaxial Test Specimens

	Zircaloy-2		Zircaloy-4
Source	AMAX Specialty Metals, Akron, New York		Teledyne Wah Chang, Albany, Oregon
Composition of As-received Material	Sn	1.49%	1.55%
	Fe	0.13%	0.225%
	Cr	0.10%	0.105%
	Ni	0.04%	< 35 ppm
	Al	< 35 ppm	46 ppm
	B	< 0.25 ppm	0.2 ppm
	C	110 ppm (111 ppm*)	145 ppm (120 ppm*)
	Cd	< 0.25 ppm	< 0.2 ppm
	Co	< 10 ppm	< 10 ppm
	Cu	< 25 ppm	16 ppm
	Hf	82 ppm	41 ppm
	H	12 ppm (31 ppm*)	10 ppm (9 ppm*)
	Mn	< 25 ppm	< 25 ppm
	N	38 ppm (27 ppm*)	33 ppm (25 ppm*)
	O	1420 ppm (1365 ppm*)	1100 ppm (1045 ppm*)
	Si	64 ppm	50 ppm
	Ti	< 20 ppm	< 25 ppm
	W	< 50 ppm	< 25 ppm
	U	0.025 ppm	1.0 ppm
Grain size	~11 μ m		~15 μ m
Thickness	0.027 and 0.032 in.		0.025 in.
Specification	ASTM Z-59		ASTM B352-67
Heat Treatment	Cold rolled and annealed		Cold rolled and vacuum annealed

* ANL analysis results.

3. Oxidation and Homogenization of Zircaloy Specimens

The high-temperature oxidation of Zircaloy results in the formation of a ZrO_2 oxide layer on α -phase Zircaloy at temperatures below the α - β transition temperature of $\sim 860^\circ\text{C}$ and a $\text{ZrO}_2/\alpha/\beta$ composite at temperatures above this transition. Thus, the test-specimen production scheme essentially consists of two parts: oxygen charging to a desired concentration and homogenization by high-temperature annealing to eliminate both the phase boundaries and concentration gradients. A Sieverts apparatus for the purpose of oxygen charging and two high-vacuum annealing furnaces for homogenization have been modified for use on this project.

a. Oxygen Charging and Homogenization Furnace

A modified Sieverts apparatus with a Marshall furnace that has a 2.5-in.-dia, 18-in.-long Pt-30% Rh heating element and a 10-in.-long

uniform temperature zone is being used for charging oxygen into the Zircaloy test specimens. A vacuum in the 10^{-6} Torr range can be achieved at temperatures up to $\sim 1300^{\circ}\text{C}$ with the diffusion pump and the liquid-nitrogen trap. Pt-Pt 10% Rh thermocouples, calibrated against the melting points of aluminum and copper, are used for temperature measurements and control sensing. The temperature-sensing thermocouple was placed at the outer surface of the alumina muffle tube, and a direct temperature reading was displayed on a strip-chart recorder. With a combination of a silicon-controlled rectifier power supply and a Digiset null detector, it was possible to stabilize temperature within $\pm 5^{\circ}\text{C}$ up to 1200°C . Specimens were placed inside a recrystallized, impervious alumina muffle tube, which was connected to the pumping and gas-handling system by a standard tapered joint with a silicone sleeve. The Zircaloy specimens were suspended from a 40-mil Pt-40% Rh wire.

Two high-temperature high-vacuum Centorr furnaces for homogenization of the Zircaloy specimens were calibrated and operated at temperatures up to 1600°C . Both systems have tungsten heating elements and tantalum heat shields that can operate in vacuum, inert-gas, or reducing atmospheres. Uniform temperatures can be maintained over half the length of the 2.5-in.-dia, 5-in.-long and 3-in.-dia, 8-in.-long heat zones. The temperatures are measured with Pt-10% Rh thermocouples and compared with optical-pyrometer and thermal-converter readings. At 1400°C , the temperature could be maintained constant within $\pm 10^{\circ}\text{C}$ for up to 30 h. Rapid heating and cooling of the specimens can be achieved; however, it takes ~ 15 min before the power level and temperature stabilize at 1400°C in the automatic control mode. Temperature readings are recorded on a strip-chart recorder.

b. Oxidation Procedure

The mechanical-property test specimens are degreased in toluene and acetone, dried, and then weighed on a microbalance. Six to ten specimens are placed inside the alumina muffle tube and the system is evacuated to 10^{-6} Torr. The specimens are heated to the charging temperatures given in Table III.2, and a measured quantity of a 1:3 oxygen-helium gas mixture is admitted to the reaction tube through a needle valve. The pressure is increased from $\sim 10^{-2}$ Torr at the onset of the reaction to ~ 2 Torr in ~ 30 s to prevent an appreciable temperature increase in the specimens. After this initial period, the gas pressure is increased to 500 Torr, i.e., an oxygen partial pressure of 125 Torr. In a few instances when the gas-admission rate was too high, an appreciable temperature increase was recorded for ~ 30 s; however, autoignition did not occur.

After the desired period of oxygen charging, the specimens are cooled and the weight gain is determined. The variations of weight gains between the specimens in the same group are within $\pm 4\%$, as indicated by the results in Fig. III.1. No reaction between the Pt-40% Rh specimen suspension wires and the Zircaloy was observed at 900 or 1000°C .

TABLE III.2. Summary of Oxidation and Homogenization Conditions for the Zircaloy Test Specimens*

Matrix Phase	C (wt. %)	T _c (°C)	t _c (min)	T _h (°C)	t _h (h)
β	0.2	900	1.5	1100	30
β	0.3	"	2.0	1200	20
β	0.4	"	3.0	1200	20
β	0.5	"	4.0	1200	20
β	0.6	"	5.4	1300	15
β	0.8	"	8.0	1300	15
β	1.0	"	11.0	1400	6
α + β	1.0	900	11.0	1300	20
"	"	"	"	1200	20
"	"	"	"	1100	30
"	"	"	"	1000	30
"	"	"	"	950	40
α + β	0.7	900	7.0	1100	20
"	"	"	"	1000	30
"	"	"	"	950	40
α + β	0.3	900	2.0	950	40
"	"	"	"	900	60
α	1.0	900	10	800	120
"	1.5	1000	4	850	100
"	2.0	1000	6	900	60
"	2.5	1000	12	1000	24
"	3.0	1000	15	1000	24
"	3.5	1100	30	1100	24
"	4.0	1100	13	1200	20
"	5.0	1100	22	1300	15
"	6.0	1100	33	1300	15
"	6.7	1200	18	1300	15

*C = oxygen concentration in wt%; T_c = oxygen charging temperature; t_c = oxygen charging time; T_h = homogenization temperature; and t_h = homogenization time.

Fig. III.1

Variation of the Average Oxygen Concentration Obtained from Weight-gain Data for 27-mil-thick Zircaloy-2 Specimens from Several Oxidation Runs. Neg. No. MSD-60893.

c. Homogenization Procedure

The oxygen-charged specimens are sealed in a 2.25-in.-dia, 5-in.-long tantalum capsule by electron-beam welding in a 10^{-5} -Torr vacuum and then annealed in either of the Centorr furnaces. The tantalum capsule acts as an effective getter in removing any reactive impurities in the residual vacuum inside the capsule. Specimens that were oxidized at 1000°C for 30 min to an average oxygen concentration of 3.6% and then homogenized for 25 h at 1000°C showed a weight gain corresponding to only 0.012% oxygen. The Pt-40% Rh wire used to suspend the Zircaloy specimens was found to be satisfactory at temperatures up to 1200°C.

At 1400°C and above, the wire forms a low-melting eutectic with Zircaloy and melts during the homogenization anneal. The Pt-10% Rh wire was satisfactory for temperatures up to 1400°C and 30 h of annealing. Above 1400°C a significant reaction occurs with either Zircaloy or tantalum and platinum, Pt-10% Rh, Pt-40% Rh, Mo, and W-Re alloys. However, rhenium was found to be satisfactory up to 7 h at 1500°C. In view of this, homogenization at 1400°C will be limited to the production of β -phase Zircaloy with 1% oxygen, which requires the high temperature because of phase-equilibrium considerations.

Homogenization of 0.8% oxygen specimens at 1400°C and 4 h showed no appreciable hardness gradient across the thickness, indicating satisfactory homogenization. It is noted that, according to the phase diagram reported by Savitskii et al.,¹ Zr-Re forms a eutectic at ~26 wt % Zr composition, which melts at ~1600°C. From our information obtained by homogenization treatments and the subsequent hardness profile measurements, a rather conservative estimate of required annealing temperature and time is given in Table III.2 for each set of conditions. It is possible to approximate the necessary time for homogenization from an equilibrium phase-boundary model, which will be discussed later. A computer program for these calculations will be used to reduce the conservatism of homogenization times.

d. Metallography and Specimen Characterization

After oxygen charging or the homogenization treatment, it is necessary to characterize the specimen both in terms of the phase-boundary locations and the oxygen-concentration gradient. Oxidized specimens with ZrO_2/α and α/β boundaries were sectioned by a carborundum wheel or an electron-beam technique, cold-mounted, polished in a vibratory polisher, and etched in a 50% distilled H_2O :45% HNO_3 :5% HF solution to observe the phase boundaries and grain structure. Anodization of the specimens in a solution containing 60-ml absolute ethanol:35-ml H_2O :20-ml glycerine:10-ml lactic acid (85%):5-ml phosphoric acid (85%):2-g citric acid was also used to develop the structure for metallographic examination. The specimen color and the current drop provided convenient criteria for adequate anodization.²⁻⁴ In

most cases, polarized light was required to obtain satisfactory contrast. The optimum procedures for specimen preparation, involving chemical etching and/or anodizing, were highly dependent upon the oxygen concentration in the alloy, the phase distributions, and whether the specimen was Zircaloy-2 or -4. Figure III.2 is a micrograph of a typical $\text{ZrO}_2/\alpha/\beta$ composite specimen obtained after oxygen charging. Figures III.3 and III.4 are photomicrographs of a partially homogenized and a completely homogenized Zircaloy-2 specimen, respectively. Martensite plates are visible in Fig. III.4 and in the central transformed $\beta \rightarrow \alpha$ -region in Fig. III.3. The latter figure also indicates the presence of cracks in the large-grain α -region of the specimen.

Fig. III.2

Photomicrograph of a Zircaloy-2 Specimen Showing the Oxide, α , and Martensitically Transformed $\beta \rightarrow \alpha$ Regions after Oxidation in an Oxygen-Helium Gas Mixture for 10 min at 1000°C. Polarized light. Mag. 316X. Neg. No. MSD-60962.

Fig. III.3

Photomicrograph of a Zircaloy-2 Specimen Oxidized under the Conditions in Fig. III.2 and then Homogenized in Vacuum for 20.6 h at 1300°C. The diffusion anneal resulted in dissolution of the oxide layer, formation of large α -grains with cracks, and a martensitically transformed $\beta \rightarrow \alpha$ region. Polarized light. Mag. ~49X. Neg. No. MSD-60886.

Fig. III.4

Photomicrograph of a Zircaloy-2 Specimen Oxidized at 900°C for 2 min, Corresponding to an Oxygen Uptake of 0.45 wt %, and Homogenized for 18.9 h at 1385°C. Martensite plates of different orientations formed from the homogeneous β -phase during furnace cooling. Polarized light. Mag. ~43X. Neg. No. MSD-60887.

To determine the presence of oxygen concentration gradients in the specimens, microhardness measurements were made using a Leitz hardness tester. The microhardness was found to be independent of load, in the range of from 25 to 500 g, and the Vickers hardness data were judged to be more reliable than the Knoop measurements. Both were calibrated against standard hardness materials. Hardness measurements in the α -phase in the immediate vicinity of the ZrO_2/α phase boundary frequently produced cracks that resulted in nonreproducible hardness values. For the homogenized specimens, the absence of phase boundaries was verified by metallographic observation and the hardness profile across the thickness. Figure III.5 shows typical microhardness indentations on an oxidized $\text{ZrO}_2/\alpha/\beta$ composite specimen and on unoxidized homogeneous Zircaloy-2 material. The measurements were made at $\sim 25\text{-}\mu\text{m}$ intervals across the specimens.

Fig. III.5. Photomicrograph of Microhardness Indentations on (a) Zircaloy-2 Specimen Oxidized for 30 min, Indicating the Presence of an Oxygen Concentration Gradient near the Surface (Mag. 86X), and (b) As-received Zircaloy-2 with No Hardness Gradient across the Specimen (Mag. $\sim 74\text{X}$). The Knoop and Vickers hardness data were obtained with a 100-g load. Neg. No. MSD-60888 and -60889.

4. Mechanical-testing Facilities

An Instron, model TTDML, machine is presently being used for the tensile testing. This machine is equipped with a Brew furnace that can operate at temperatures up to 2000°C at a 10^{-6} -Torr vacuum. The specimen is held between grips made of TZM alloy. At high temperatures, the fracture loads for the Zircaloy specimens are small. To record these small loads more accurately, a low-capacity load cell with high sensitivity is presently being purchased.

To conduct the extensive mechanical testing on a full-time basis, a new Instron machine (Model 1125) has been purchased. This machine will

also have a recorder with a faster response. The heating and cooling rate of the Centorr furnace, to be installed on the new Instron, will be controlled by a Data-Trak programmer.

5. Test Matrix for Uniaxial Specimens

The matrix for the uniaxial tensile specimens is given in Table III.3. Although most of the tests will be performed on Zircaloy -4, a sufficient number

TABLE III.3. Test Matrix for Determining the Effect of Oxygen Concentration and Strain Rate on the Tensile Properties of Zircaloy-2 and -4

1. β -phase Zircaloy						
Test Temp., °C	Oxygen Conc., wt%			Strain Rate, s ⁻¹		
1400	0.1	0.5	1.0	Extensive testing at 10 ⁻¹ and 10 ⁻⁴		
1300	0.1	0.4	0.8			
1200	0.1	0.3	0.6	Scoping tests at 10 ⁻² and 10 ⁻³		
1100	0.1	0.3	0.5			
1000	0.1	0.2	0.3	Strain-rate change tests between 10 ⁻² and 10 ⁻³		
900	0.1					
Number of Specimens: 190 with 20% duplication						
2. $\alpha + \beta$ phase Region [†]						
Test Temp., °C	Oxygen Conc., wt%			Strain Rate, s ⁻¹		
1300			1.0	Extensive testing at 10 ⁻¹ and 10 ⁻⁴		
1200			1.0			
1100		0.7	1.0			
1000		0.7	1.0			
950	0.3	0.7	1.0			
900	0.3	0.7				
Number of Specimens: 130 with 20% duplication						
* A cooling rate of 2 and 15°C/s from 1400°C to test temperature will be used.						
3. α -phase Zircaloy						
Test Temp., °C:	RT, 200, 400, 600, and 800					
Oxygen Conc., wt%:	0.1, 0.5, and 1.0					
Strain Rate, s ⁻¹ :	10 ⁻¹ and 10 ⁻⁴					
Number of Specimens:	90 with 20% duplication					
4. ZrO ₂ /α/β-phase Composite Specimens ^{**}						
Test Temp., °C	Fractional Thickness of β-Phase, F _w				Strain Rate, s ⁻¹	
1400	0.1	0.3	0.5	Extensive testing at 10 ⁻² and 10 ⁻⁴		
1200	0.1	0.3	0.5			
1000	0.1	0.3	0.5			
800		0.3	0.5			
600		0.3	0.5			
400		0.4	0.5			
200			0.5			
			0.6			
			0.7			
			0.7			
			0.7			
Number of Specimens: 90 with 50% duplication						

* A cooling rate of 2 and 15°C/s from 1400°C to test temperature will be used.

** Specimens produced at 1200°C and cooled at 2°C/s to test temperature.

of scoping tests will be made on Zircaloy-2 to establish if any important differences exist in the high-temperature mechanical behavior of the two materials. Test matrices have also been developed for four-point-bend specimens of α -phase Zircaloy, containing between 1.0 and ~6.7 wt % oxygen, and for biaxial tube specimens with oxygen concentrations and oxygen gradients that are similar to those given in Table III.3 for the uniaxial test specimens.

B. Results

1. Mechanical Properties of As-received Zircaloy-2

Figures III.6 and III.7 show the engineering stress-strain curves for Zircaloy-2 specimens fractured at the strain rate of $3.3 \times 10^{-3} \text{ s}^{-1}$. At strains larger than 0.1, these curves do not truly represent the stress-strain state of the deforming material due to the significant changes in the cross-sectional area and the gauge length. Therefore, it was suggested⁵ that the two axes in Figs. III.6 and III.7 be labeled normalized load and normalized elongation, which may be a better physical representation of the experimental measurements. To maintain consistency in the dimensional units and conventional nomenclature, the axes in these figures are labeled as stress and strain. The stress was based on the original cross-sectional area, and the strain was calculated from the original gauge length. The maximum load point (corresponding to the uniform strain) on all these curves occurred at a strain smaller than 0.1. Therefore, true-stress and true-strain calculations beyond the uniform strain need a necking correction to account for the triaxial state of stress.

Fig. III.6

Engineering-stress/Engineering-strain Curves for As-received Zircaloy-2 Specimens Deformed at Temperatures between 750 and 950°C. Neg. No. MSD-60897.

Fig. III.7

Engineering-stress/Engineering-strain Curves for As-received Zircaloy-2 Specimens Deformed at Temperatures between 1000 and 1400°C. Neg. No. MSD-60895.

To apply the necking correction, neck profile measurements during the test are necessary. Such measurements were not possible with the present equipment because of the high-temperature/high-vacuum environmental chamber. The measurement of the fracture area to calculate the true fracture stress also has a number of practical difficulties that result from excessive grain growth. These include the unevenness of the specimen surface, which makes it difficult to measure the thickness, double-neck formation, and the small specimen thickness at the point of fracture.

To obtain true-stress-true-strain curves for a limited number of test conditions, the following types of tensile tests are planned. Specimens will be strained to different points on the load-elongation curve, unloaded, and metallographically polished to measure the minimum area. From the minimum area, gauge-length profile, and the load value at the point of unloading, the true stress and true strain will be calculated. Even with short hold times at the testing temperature, considerable grain growth was observed in the specimens tested above 1000°C.

A linear-intercept grain-size estimation in the gauge length of the fractured specimens revealed that, up to 1000°C, the grain size is close to the as-received grain size of 11 μm . At higher temperatures, grain-size estimates are as follows: 1100°C (440 μm), 1200°C (600 μm), 1300°C (750 μm), and 1400°C (850 μm). Thus, the stress-strain curves in Fig. III.7 correspond to different grain sizes. Grain coarsening during the simulated LOCA situation has been observed in Zircaloy-2.⁶ However, the effect of such grain growth on the mechanical properties of Zircaloy has not been investigated. The tensile testing of oxidized β -Zircaloy in this program will also involve grain coarsening (possibly to a lesser extent). To isolate the effects of grain size and oxygen concentration on the tensile properties of Zircaloy, constant temperature-constant strain-rate tests are presently under way to investigate the effect of grain size on the tensile properties of as-received Zircaloy-2.

The temperature dependence of the 0.2% offset yield stress and ultimate tensile strength is plotted in Fig. III.8. Although both the yield stress and the ultimate tensile strength decrease continuously with an increase in temperature, a hump occurs in both curves near 900°C. The variations of the uniform strain, total strain, and necking strain (from the maximum load point to the fracture point) with temperature are shown in Fig. III.9. Note that below 1100°C, the uniform elongation is almost zero and that necking elongation almost completely accounts for the total elongation. Another significant feature of Fig. III.9 is the minimum in the necking strain and total strain at 950°C.

The strain-rate sensitivity parameter, m , is defined by the relation

$$(\sigma_2/\sigma_1) = (\dot{\epsilon}_2/\dot{\epsilon}_1)^m,$$

where σ_1 and σ_2 are the flow stresses at strain rates $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$, respectively. When the flow-stress response following a strain-rate change shows a transient (Fig. III.10), two values of strain-rate sensitivity can be calculated.⁷ The

instantaneous value m' corresponds to the flow-stress change $(\sigma_2' - \sigma_1)$, i.e., $m' = [\log(\sigma_2'/\sigma_1)]/[\log(\dot{\epsilon}_2/\dot{\epsilon}_1)]$; and the extrapolated steady-state value m'' corresponds to the flow-stress change $(\sigma_2'' - \sigma_1)$, i.e., $m'' = [\log(\sigma_2''/\sigma_1)]/[\log(\dot{\epsilon}_2/\dot{\epsilon}_1)]$. The variation of the strain-rate sensitivity of Zircaloy-2 with strain at 900 and 1200°C is shown in Fig. III.11. These data points were obtained by changing the strain-rate between $3.3 \times 10^{-4} \text{ s}^{-1}$ and $3.3 \times 10^{-3} \text{ s}^{-1}$. Note that during the first several percents of strain, m decreases, and this decrease is more significant at the higher temperature (1200°C). At 900°C, necking starts at ~2% strain, and, therefore, the data points beyond 2% strain in the 900°C curve in Fig. III.11 are not completely accurate.

Fig. III.8

Effect of Temperature on the 0.2% Yield Stress and the Ultimate Tensile Strength of As-received Zircaloy-2. Neg. No. MSD-60902.

Fig. III.9

Effect of Deformation Temperature on the Uniform, Necking, and Total Strains for Zircaloy-2 Specimens. Neg. No. MSD-60901.

Fig. III.10

Schematic Stress-Strain Curve for a Strain-rate Change Experiment. When flow stress transients appear following a strain-rate change, two values of the strain-rate sensitivity parameter can be calculated. Neg. No. MSD-60892.

Fig. III.11

Variation of the Strain-rate Sensitivity Parameter with Plastic Strain for Zircaloy-2 at 900 and 1200°C. Neg. No. MSD-60903.

The presence of transients following a strain-rate change at 900 and 1200°C, the ductility minimum at 950°C (Fig. III.9), and the hump in the UTS and yield stress versus temperature diagram near 900°C (Fig. III.8) imply a dynamic strain-aging effect at these high temperatures in Zircaloy-2. The work-hardening rate of Zircaloy-2 is presently being analyzed to check the extent of dynamic strain aging. These dynamic strain-aging manifestations have been reported for α -zirconium⁷ and α -Zircaloy-2,⁸ although at a much lower temperature.

Several scoping tests have been made at initial strain rates between 10^{-4} and 10^{-5} s^{-1} . At 900°C, the Zircaloy specimens exhibited superplastic behavior characterized by a small (practically zero) uniform elongation, quite large necking elongation (and therefore a large total elongation), and a large value (>0.2) for the strain-rate sensitivity parameter. The macrophotograph in Fig. III.12 compares a Zircaloy-2 specimen deformed at 900°C ($\dot{\epsilon} = 6.6 \times 10^{-5} \text{ s}^{-1}$) and an undeformed specimen. Even after deformation to a strain of 220%, no visible concentrated neck occurs in the gauge section of the specimen. A considerable strain in the width and thickness directions is also present. As seen in Fig. III.12 (deformed specimen), the strain in the width direction is $\sim 50\%$ in the center of the gauge length. At the same point the strain in the thickness direction was measured as $\sim 40\%$. A similar behavior was observed at the strain rate of $3.3 \times 10^{-4} \text{ s}^{-1}$. The factors responsible for this superplastic behavior are probably the fine grain size, two-phase structure, and possibly deformation-induced transformation. As stated earlier, grain coarsening occurs above 1000°C. At 900°C, on the other hand, some grain-size refining occurs during deformation. For example, in the gauge section of the specimen deformed at 900°C, the average grain size is $\sim 7 \mu\text{m}$. The undeformed shoulder region of the same specimen shows a grain size of $11 \mu\text{m}$, the same as the as-received condition. A similar effect was observed

Fig. III.12

Comparison between a Zircaloy-2 Specimen Strained under Superplastic Conditions (i.e., 900°C, $\dot{\epsilon} = 6.6 \times 10^{-5} \text{ s}^{-1}$) and an Undeformed Specimen. Neg. No. MSD-60891.

by Backofen et al.⁹ in the deformation of a two-phase Ti-6Al-4V alloy. A metallographic examination of our 900°C specimens also revealed evidence of a two-phase structure. Superplasticity was reported⁹ for fine-grain Zircaloy-4 deformed in the transformation region. To assess the importance of different deformation mechanisms of superplasticity (viz.,¹⁰ vacancy creep, grain-boundary sliding, and dislocation creep), tensile tests are being performed at various strain rates on Zircaloy specimens with a range of grain sizes.

A metallographic procedure involving anodizing²⁻⁴ was used to study the microstructure under the polarized light. Figure III.13 shows a typical structure representing two phases (α and β) cooled from 950°C. Below the transformation temperature, the high-temperature β -phase transforms to martensitic α -phase.

Fig. III.13

Room-temperature Microstructure of a Zircaloy-2 Specimen with Two Types of α Grains Representing a Two-phase Structure at High Temperature. The acicular martensitic α -grain results from quenching the high-temperature β Phase. Mag. 500X. Neg. No. MSD-60890.

2. Mathematical Analysis of the Oxidation and Homogenization Kinetics

To provide more accurate information on the oxidation and homogenization kinetics for specimen preparation above the α/β transition

temperature, a computer program was developed for a finite-thickness specimen geometry that takes into account phase-boundary migration during the oxidation and homogenization processes. The analysis is based upon diffusion-controlled oxygen migration in which the equilibrium oxygen concentrations are assumed for the ZrO_2 , α , and β phases at the ZrO_2/α and α/β phase boundaries. Although the details of the analysis will be reported at a later date, Fig. III.14 shows the average oxygen concentration in 27- and 30-mil-thick specimens as a function of the oxidation time at temperatures between 900 and 1420°C. The data points were obtained from gravimetric measurements on 27-mil-thick Zircaloy-2 specimens at 900 and 1000°C. The location of the α/β phase boundary as a function of oxidation time and temperature is shown in Fig. III.15. The data points at 900 and 1000°C were obtained from

metallographic measurements of the oxide and α -phase thicknesses on the 27-mil-thick Zircaloy-2 specimens. A Pilling-Bedworth ratio of 1.56 was used to locate the original surface from the observed oxide thickness. The original surface of the unoxidized specimen is the zero reference location on this figure. The thickness of the oxide layer was found to be in agreement with the expression

$$E_o(t_c) = 2R\gamma_o(D_o t_o)^{1/2},$$

where $R = 1.56$, $\gamma_o = 0.58$, t_c is the oxidation time, and E_o and D_o are the oxide layer thickness and the diffusion coefficient for oxygen in the oxide phase, respectively. The results in Fig. III.15 are in good agreement with metallographic measurements of phase-boundary locations on oxidized Zircaloy specimens reported by Meservey and Herzel⁶ and Hobson and Rittenhouse¹¹ for moderate degrees of oxidation [i.e., $(D_\alpha t)^{1/2}/L < 1.5$, where D_α is the diffusivity of oxygen in the α phase, t is the oxidation time, and L is the specimen thickness]. For larger degrees of oxidation corresponding to longer times at the higher temperatures, the analysis predicts a considerable deviation from parabolic oxidation kinetics.

Fig. III.14

Effect of Oxidation Time and Temperature on the Computed Average Oxygen Concentrations in 27- and 30-mil-thick Zircaloy-2 Specimens. The data points were obtained from gravimetric measurements on 27-mil-thick specimens at 900 and 1000°C. Neg. No. MSD-60900.

Fig. III.15

Calculated α - β Phase-boundary Penetration during Zircaloy Oxidation, Indicating Increasing Deviation from Parabolic Kinetics at the Higher Temperatures and Longer Oxidation Times. The original surface of the unoxidized specimen is the zero reference location. Data points were obtained from metallographic measurements of the oxide and α -phase thicknesses on 27-mil Zircaloy-2 in which a Pilling-Bedworth ratio of 1.56 was used to locate the original surface from the observed oxide thickness. Neg. No. MSD-60899.

Initial calculations of the homogenization kinetics for a specimen oxidized for 30 min at 1000°C and then annealed at this temperature to produce α -phase Zircaloy with 3.6 wt % oxygen is shown in Fig. III.16. Oxygen profiles that describe the latter stages of the homogenization process are not available at this time. The curves in Fig. III.16 indicate that the oxygen concentration gradient in the β -phase region of the specimen practically disappears during the early stages of homogenization as a result of the high diffusion coefficient for oxygen in β compared with that in the ZrO_2 and α phases. Based upon the kinetic analysis for the oxidation and homogenization processes, it should be possible to optimize the oxidation and annealing conditions to produce composite $\text{ZrO}_2/\alpha/\beta$ mechanical-test specimens with desired compositions and phase distributions.

Fig. III.16

Computed Oxygen Concentration Profiles across a Zircaloy Specimen. Curve (A) after oxidation for 30 min at 1000°C and curves (B) and (C) after homogenization in vacuum for 20 and 50 min at 1000°C, respectively. The oxygen concentrations in the ZrO_2 , α , and β phases at the ZrO_2/α and α/β phase boundaries are based upon the zirconium-oxygen phase diagram. Neg. No. MSD-60898.

after the homogenization treatment. This specimen was oxidized for 30 min at 1000°C and then annealed for 25 h at this temperature. Curve B corresponds to the calculated oxygen concentrations that were converted to hardness readings using Fig. III.17. The phase-boundary locations were also computed from the kinetic analysis for the finite-thickness specimen geometry. Reasonable agreement between the microhardness measurement and the "semi-theoretical" profile was obtained in the β -phase region.

Microstructures of the pseudobinary Zircaloy-2 and -4 alloys containing oxygen indicate that they are considerably different than those expected from a consideration of the binary zirconium-oxygen system. Since the

Since it was expected that microhardness measurements would provide useful information concerning the oxygen concentration profiles and also an indication that homogenization had been achieved, a plot of room-temperature hardness versus oxygen concentration was developed for Zircaloy-2. The results in Fig. III.17 indicate that the hardness measurements are sensitive to oxygen concentration up to ~1.5 wt %.

Figure III.18 shows the hardness profiles from a Zircaloy-2 specimen before and after oxidation and

composition and distribution of the phases (i.e., microstructure) are important in the interpretation of the results of mechanical-property investigations and are also required in models for the isothermal and transient oxidation behavior, the scope of the present work will include the determination of the equilibrium phase boundaries in the Zircaloy-oxygen system. In view of the considerable grain growth that has been observed in Zircaloy at high temperatures, a systematic study of the effect of oxygen concentration and temperature on the kinetics of this process is also in progress.

Fig. III.17

Relationship between Room-temperature Vickers Microhardness Number (100-g load) and Oxygen Concentration in Zircaloy-2. Neg. No. MSD-60894.

Fig. III.18

Vickers Microhardness Profiles across Zircaloy-2 Specimens. (A) Values for an as-received specimen with 0.135 wt % oxygen, (B) corresponds to a specimen oxidized for 30 min at 1000°C, and (C) represents specimen (B) after homogenization for 25 h at 1000°C. The broken-line curve for specimen (B) was obtained by converting the oxygen concentration profile computed from the equilibrium phase-boundary model into hardness values using the data in Fig. III.17. The ZrO_2 , α , and β phase boundaries prior to homogenization are indicated by vertical lines. Neg. No. MSD-60896.

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