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HIGH-PRESSURE TEST LOOP DESIGN AND APPLICATION

by

**R. D. BURNETTE, J. N. GRAVES, P. G. BLAIR,
and N. L. BALDWIN**

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ABSTRACT

A high-pressure test loop (HPTL) has been constructed for the purpose of performing a number of chemistry experiments at simulated HTGR conditions of temperature, pressure, flow, and impurity content. The HPTL can be used to develop, modify, and verify computer codes for a variety of chemical processes involving gas phase transport in the reactor. Processes such as graphite oxidation, fission product transport, fuel reactions, purification systems, and dust entrainment can be studied at high pressure, which would largely eliminate difficulties in correlating existing laboratory data and reactor conditions.

1. INTRODUCTION

Within the high-temperature gas-cooled reactor (HTGR), the graphite, metallic, and ceramic components are exposed to high temperatures (approximately 300° to 1000°C), high helium pressures [up to 7.2 MPa (1050 psi)], and high (turbulent) gas flow rates (Reynolds numbers up to approximately 100,000). Many chemical processes that occur in the reactor involving gas transport phenomena are influenced by these three main factors. Examples of possible gas-transport-controlled phenomena are graphite oxidation; carbon deposition; fuel hydrolysis; boron oxidation and transport; dust entrainment; sorption and desorption processes in the purification train; and fission product release, diffusion, vaporization, plateout, and liftoff. Heretofore, most chemical research in support of the HTGR has neglected the latter two factors, pressure and flow, because no high-pressure facility was available. Such experiments were typically performed in vacuum or at ambient pressure using once-through flow systems. While the results of those tests have proved useful, in some cases no direct correlation between reactor conditions and the existing data has been possible. Rather, assumptions based on theory have been made as to the effects of high pressure. Many of these assumptions have not been verified experimentally.

At General Atomic, the high-pressure test loop (HPTL) has been constructed, is now in operation, and is available for a variety of important chemistry experiments. In concept, the HPTL offers numerous advantages over once-through experiments since, under loop conditions, HTGR fluid dynamics can be simulated where long-term steady-state reactant and product concentrations are achievable. In addition, the effective molecular transport of active species across the boundary layer and in porous media in the reactor can be duplicated (excluding possible radiation effects). Accordingly, HPTL experiments will complement existing data and provide the necessary refinement and verification of current predictions of chemical reactions and transport phenomena obtained from computer codes.

2. TEST LOOP DESIGN

A block diagram depicting the HPTL flow system is shown in Fig. 1. The loop consists of a series-connected single-stage diaphragm compressor, a regenerative heat exchanger, and a test chamber. The heat exchanger, test chamber, and heating elements are contained within an insulated pressure vessel (Fig. 2). The loop is designed to provide continuous flows up to 19 standard l/s (40 scfm) at 6.9 MPa (1000 psig) total pressure. The graphite sample configuration in Fig. 3 is typical of planned graphite oxidation tests. Flow velocity through the central channel (10-mm diameter) of the test specimen is calculated to be 1500 cm/s at 1000°C with a Reynolds number of 5000. At these conditions, the mass transfer coefficient (H_2O in helium) in the Fort St. Vrain reactor core support block channel is duplicated. A cylindrical test section 150 mm long and 80 mm inside diameter is available to accommodate candidate graphite samples or other selected materials at uniform test temperatures up to 1000°C. Gas impurities are controlled at desired levels by a combination of continuous injection of impurity gas and helium, bypass purification, and continuous bleed to a monitoring system.

Pressure, flow, and temperature are measured using conventional pressure gauges and transducers, electronic mass flowmeters, and thermocouples. Data are collected and recorded by use of a 40-channel data logger. Loop components and specifications are given in Table 1.

The pressure vessel consists of a central Inconel chamber in an outer steel jacket. The annular space around the central chamber is insulated with a high-purity fibrous ceramic material. Super Kanthal heater elements, capable of prolonged operation at 1200°C, are located within the central Inconel chamber and positioned around the test sample. The test chamber and insulated annulus are interconnected to equalize the pressure across the Inconel tube wall. Differential pressure between the two chambers is monitored by a Bourdon-type pressure gauge and a differential pressure

Fig. 1. High-pressure test loop flow diagram

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Fig. 2. High-pressure test loop pressure vessel showing thermocouple leads

Fig. 3 . Details of sample chamber

TABLE 1
HIGH-PRESSURE TEST LOOP COMPONENTS AND SPECIFICATIONS

Test chamber parameters

Dimensions	150 mm long, 80 mm inside diameter
Temperature	1000°C maximum
Volumetric flow	1.2 l/s (2.5 ft ³ /min) at 1000°C and 5.2 MPa
Mass flow	2.3 g He/s
Operating pressure (max)	7 MPa (1000 psig)
Gas velocity	15 m/s (50 ft/s)
Reynolds number	5000
Sample central channel	10 mm inside diameter

Bypass purification system

Oxidizer trap	Cu/CuO at 300°C
Dryer trap	Ascarite, Mg(ClO ₄) ₂ at 25°C
Liquid nitrogen trap	Activated carbon at -196°C

Monitor system

Trace gas analyzer	Varian (He-ionization detector)
Hygrometer	EG&G, Model 440 (dew point)
Recorder	Linear, Model 385
Thermocouples	Gordon, dual element, Type K
Pressure transducers, differential	Viatran, Model 218 Viatran, Model 223
Mass flowmeters	Teledyne-Hastings - Raydist, Model A111KPG
Pressure gauges	Weksler, Bourdon type
Digital thermometers	Doric, Model 402A
Digital pressure indicators	Viatran
Digital flow indicators	Teledyne-Hastings - Raydist

Data acquisition system

Data logger	Doric Model 240
Data terminal	Texas Instruments, Model ASR 733

Circulating pump

Pump	Pressure Products Industries, Model 3078 (single-stage diaphragm)
Stroke	100 mm (4 in.)
Displacement	11.3 cm ³ (0.692 in. ³) at 400 rpm
Horsepower	10
Maximum flow	1140 std l/m (40 scfm) at 1000 psi
Maximum pressure	7 MPa (1000 psig)

transducer. A digital readout for the differential pressure transducer is located at the system control panel. Fourteen penetrations on the pressure vessel are used for thermocouples (seven), heater leads (two), and helium inlets/outlets to both chambers (four).

The test section, which is 324 mm long with an inside diameter of 81 mm, is contained within the central Inconel chamber. The isothermal zone is about 150 mm long, which dictates the sample length. As indicated in Fig. 3, incoming impurity-laden helium is preheated inside the vessel by first flowing through the shell side of the regenerative heat exchanger (A) and then up the chamber wall in direct contact with the heating element (H). The heated gas flows into the central channel of the graphite sample (F), which is encased in a crucible constructed of relatively nonreactive graphite (G). The gas then exits through the tube side of the regenerative exchanger, where it is cooled by the incoming gas. For the experiment depicted in Fig. 3 (graphite oxidation), where it is necessary to achieve high temperatures, much of the space in the central chamber is taken up by the heating element. For tests with lower required temperature, the heating element can be positioned outside the Inconel chamber, thereby increasing the available sample volume.

Impurities of interest are introduced into the loop by one of two systems. Steam, a potential oxidant, is injected by passing a bypass flow of helium over a water or ice reservoir. The rate of injection into the system is controlled by regulating the flow and reservoir temperature. The reservoir temperature is maintained at $\pm 0.2^\circ\text{C}$ using a commercial temperature controller. Other impurities (CH_4 , H_2 , CO , and CO_2) are introduced into the loop from premixed pressurized cylinders. The impurity concentrations of the cylinders are certified by the vendor (Matheson) and analyzed, before introduction into the loop, using the trace gas analyzer. Rates of entry are controlled by fine metering valves and mass flowmeters.

The impurity concentration is controlled at steady state by one of two bypass purification systems, shown in Fig. 4. Bypass flows from 1% to 10%

Fig. 4. High-pressure test loop bypass purification system

(of inventory) per hour are used. Gas phase impurities are removed or converted by three separate units, as shown in Fig. 4. The oxidizer trap contains an Englehardt CuO catalyst, which converts CO and H₂ to CO₂ and H₂O at temperatures of 300°C. A mixed bed drier column is composed of Ascarite (NaOH-asbestos) sandwiched between beds of magnesium perchlorate for removal of CO₂ and H₂O. The third trap is activated carbon maintained at liquid nitrogen temperatures for removal of all other species. The system is designed to function in series or parallel and in the bypass or regeneration mode. In the regeneration mode, the spare system is in service. Purification flow is measured using a mass flowmeter; temperatures are measured using Type K thermocouples. The CuO bed is heated by heating tapes. Flow and temperature data are fed directly to a data logger, which provides a continuous scan and record of these readings.

Figure 5 is a photograph of the control panel and monitoring instruments. The monitoring system consists of a trace gas analyzer, with a dual pen recorder and digital integrator, and a dew-point hygrometer. Hygrometer output is fed to a front panel meter and to the data logger. Data from the trace gas analyzer are fed to the recorder, integrator, and data logger. The impurity monitors accept a constant small bleed flow of loop gas. The hygrometer is a constant-reading dew-point device with a range of from -75° to +60°C and a sensitivity of $\pm 0.06^\circ\text{C}$.

The trace gas analyzer is equipped with an automatic sampling valve, which can be programmed for frequent (every 1/2 hour) to infrequent (daily) sampling rates. Tritium source helium ionization detectors are used in the trace gas analyzer. Detector outputs are amplified by means of an electrometer with a sensitivity of 5×10^{-3} A/mV. Detection and amplification systems provide detection capabilities for H₂, O₂, N₂, CO, CO₂, and CH₄ of less than 1 ppm with precision of $\pm 10\%$. Instrument linearity and calibration have been confirmed using exponential dilution methods and standard calibration gas mixtures supplied by Matheson.

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Fig. 5. Control panel and monitoring instruments

The data acquisition system comprises a Doric 40-channel data logger, Viatran pressure transducers, Type K thermocouples, Teledyne-Hastings - Raydist mass flowmeters, an EG&G hygrometer, and a Varian trace gas analyzer. Electrical output from each sensor or instrument is fed to a discrete channel in the data logger. The data logger is programmed to scan all channels at any convenient time interval and certain critical channels more frequently using a subscan routine. Each channel has a programmed high- and low-level alarm. Data are printed on the internal printer or on a Texas Instruments ASR data terminal in the appropriate engineering units.

Bourdon-type pressure gauges are utilized at different locations on the loop proper, and digital pressure readouts are located at the control panel. Two Doric multipoint thermometers are also located on the control panel, along with digital readouts of gas flows throughout the entire system. Thus, the operator has immediate visual access to critical information in addition to recorded data from the data logger.

3. SAFETY CONSIDERATIONS

The pressure vessel of the HPTL was designed and constructed according to the design requirements of the ASME Boiler and Pressure Vessel Code, Section VIII, Division 1. The pressure vessel support structure, designed for 25% vertical impact and 0.39 horizontal seismic base acceleration, is secured to the laboratory floor. The vessel has been hydrostatically tested and certified for continuous operation at 6.9 MPa (1000 psig) at a shell or flange outer surface temperature of 204°C. Shell temperatures far below 100°C are ensured by the internal insulation and a water-cooled jacket on the outside of the vessel. Tests have shown that even without water cooling, the vessel wall temperatures do not exceed 100°C. Water cooling was provided to ensure additional safety margin. In the event of failure of the water-cooling system, an alternate water supply is switched on. If the alternate supply fails, the furnace is automatically switched off via a flow switch in the cooling system.

Eight Type K dual-element thermocouples monitor the pressure vessel shell and flange temperatures. All thermocouples are calibrated against a National Bureau of Standards traceable reference thermocouple. One of the dual thermocouples at each location is recorded every hour by the data logger. The other thermocouple is monitored by a digital thermometer with manual pushbutton switching. Logic is built into each channel of the data logger to allow high and low visual-alarm set points for pressure and temperature as well as high and low power scram set points. Thus, the shell and flange thermocouples are set to shut off power to the furnace heater in the event shell temperatures exceed 121°C (which constitutes a second independent pressure vessel overtemperature protection system). The sample centerline temperature, Kanthal heater temperature, and vessel helium inlet and outlet temperatures are all independently monitored with Type K dual-element thermocouples. The furnace and sample temperature data logger scram points are set at $\pm 20^\circ$ of the intended sample temperature.

The HPTL system plumbing, including valves, flow indicators, and pressure transducers, is fabricated from Type 304 or Type 316 stainless steel. The main loop consists of 12.7 mm (0.5 in.) o.d. stainless steel tubing with 1.24 mm (0.049 in.) wall thickness rated for 25.5 MPa (3700 psig) at 210°C. Subsystem piping is of either 3.18 mm or 6.35 mm (1/8 in. or 1/4 in.) o.d. stainless steel tubing, and all pipe connectors are stainless steel Swagelok. Pipe runs are secured by brackets to prevent pipe whip.

The loop is protected from overpressurization by an independent relief valve set at 5.5 MPa (800 psig), which may be adjusted upward to higher pressures if required. The diaphragm compressor for circulating helium through the loop is rated for continuous service at 6.9 MPa (1000 psig) outlet pressure. It is equipped with a relief valve set at 10.34 MPa (1500 psig) and an automatic shutoff in the event of diaphragm failure. A pressure transducer at the pump inlet is set to shut off power to the pump if the pressure exceeds 5.5 MPa (800 psig) (may be adjusted higher if required). Helium flow in the loop is monitored with mass flow monitors at the pump inlet, all five chemical impurity injection points, and both loops of the chemical impurity cleanup system.

4. APPLICATIONS

4.1. GRAPHITE OXIDATION

The HPTL was originally designed to study the effect of pressure and flow on graphite oxidation, with particular regard to the change in burnoff profile at high pressure, and to changes in surface corrosion due to high flow.

Graphite oxidation can occur in the HTGR as a result of interactions between water vapor and graphite core components. Leaks from the steam generator, ingress from the water-lubricated circulator, and component outgassing during startup (initial and after refueling) are possible sources of water vapor.

Graphite oxidation can be considered to be comprised of a series of steps: (1) transport or diffusion of water vapor across the stagnant boundary layer to the surface, (2) in-pore diffusion to active sites within the graphite, (3) chemical reaction to form products CO and H₂, and (4) diffusion of products out of the graphite. In this overall process, steps 1, 2, and 4 are definitely influenced by total pressure (the gas diffusion coefficients of H₂O, CO, and H₂ are inversely proportional to pressure). At the higher temperatures, reaction rates can be so high that step 1, transport across the boundary layer, is controlling. In this case the overall rate of reaction depends solely on the rate of arrival of oxidant molecules at the surface via diffusion, which is inversely proportional to total pressure. At intermediate temperatures, the chemical reaction rate is lower and the overall process is controlled by steps 2 and 3, simultaneous in-pore diffusion and chemical reaction. In this case the overall reaction is described by the Dodson equation (Ref. 1),

$$R_T = S/V L F_1 R_s \quad , \quad (1)$$

where R_T = the total reaction rate of the sample, s^{-1} ,
 S/V = the surface-to-volume ratio of the graphite specimen, cm^{-1} ,
 L = the diffusion length given by $(D_{eff}/K_V)^{1/2}$,
 $D_{eff} = mD_{1,2}/P_T$,
 $D_{1,2}$ = the diffusion coefficient of H_2O in helium,
 m = a constant characteristic of the porosity and tortuosity of the graphite,
 P_T = total pressure, atm, and
 K_V = the volumetric reaction rate constant, s^{-1}
 $= R_S \rho RT/12 P_{H_2O}$.

The term R_S is the chemical surface reaction rate in the absence of mass transport control, s^{-1} , and F_1 is a geometrical effectiveness factor which, for studies of highly reactive graphite, is essentially 1. A useful result of these formulae where $F_1 = 1$, and assuming a first-order reaction where burnoff B is directly related to water concentration, is

$$\ln B = -x/L + \ln B_0 \quad , \quad (2)$$

where B_0 and B are the fractional weight losses at the surface and at depth x , respectively. By plotting $\ln B$ versus x , a straight line* of slope $1/L$ is obtained. The intercept B_0 divided by time is the average reaction rate R_S in the absence of mass transfer or diffusion control. From the direct measurements of L and R_S , the effective porosity/tortuosity coefficient m is obtained using the equation

$$m = \frac{L^2 R_S \rho}{12 D_{1,2} P_{H_2O}} \quad . \quad (3)$$

*Near the surface, the profile usually curves over, having a lesser slope, indicating an increased D_{eff} or m value in the highly oxidized zone. By constructing asymptotes to the curved portion, it is possible to determine m at varying burnoffs. In theory, m should approach 1 at 100% burnoff.

For the case of high temperatures or highly reactive graphites where $r_o/L \gg 50$, $F_i = 1$ (r_o = sample radius). At lower temperatures or for less reactive graphite grades, F_i may be described, for solid cylinder geometry, by

$$F_{i_s} = \frac{I_1(r_o/L)}{I_0(r_o/L)} \quad (4)$$

where r_o is the sample radius, and I_1 and I_0 are modified vessel functions of the first kind, first and zero order, respectively.

$$F_{i_h} = \frac{K_1(r_i/L) I_1(r_o/L) - I_1(r_i/L) K_1(r_o/L)}{K_1(r_o/L) I_0(r_i/L) + K_0(r_i/L) I_1(r_o/L)} \quad (5)$$

For hollow cylinder geometry, where K_0 and K_1 are modified Bessel functions of the second kind, zero and first order, respectively, r_o is the overall sample radius, and r_i is the central hole radius.

Hollow cylindrical geometry is used in the oxidation tests (Fig. 3.). The test gas is forced to flow through the central 10-mm-diameter circular channel at velocities up to 15 m/s, which is roughly equivalent to the gas velocity in the HTGR core. The mass transfer coefficient k for these experimental conditions is calculated using the equation

$$k = Sh \frac{D_{1,2}}{d} \quad (6)$$

where $Sh = 0.023 (Re)^{0.83} (Sc)^{0.44}$ for $2000 < Re < 35,000$ (Sherwood number),

$Re = du/\nu$ (Reynolds number),

$Sc = \nu/D_{1,2}$ (Schmidt number),

$u = 4Q/\pi d^2$ (gas velocity),

d = channel diameter, cm,

l = channel length, cm,

Q = volumetric flow rate, cm^3/s , and
 ν = kinematic viscosity.

The mass transport coefficient k' for water vapor in graphite for the case of simultaneous diffusion and reaction is

$$k' = (D_{\text{eff}} K_V)^{1/2} \quad , \quad (7)$$

where K_V is the volumetric reaction rate coefficient defined above. The term k/k' is the ratio of mass transfer to chemical reaction. Using estimated values of K_V and D_{eff} for PGX graphite, the data in Table 2 were calculated. It is seen that in the large ducts of the Fort St. Vrain support blocks, $k/k' = 1.1$, which means that at steady state the concentration of water at the surface is about one-half that of the bulk gas, due to depletion. This condition is closely simulated in the HPTL experiment.

4.2. OTHER APPLICATIONS

Other experiments which are planned include an integral fuel hydrolysis test in which the effects of moisture depletion via oxidation of graphite and fuel matrix can be assessed. Since high pressure has the effect of retarding transport of moisture through the graphite, less moisture in the fuel zone is predicted, with a consequent decrease in the rate of fuel hydrolysis. Such predictions need to be verified by integral tests in the HPTL. The tests can be used to modify or verify the hydrolysis/oxidation codes, OXIDE (Ref. 2) and HYDROBURN (Ref. 3).

Although the release and diffusion of the gaseous fission products xenon, krypton, and iodine would be affected by pressure, it is possible that the transport of cesium through graphite may also be affected. A simple test at high pressure would resolve this question. In addition, vaporization of cesium and strontium from graphite may be affected by the turbulent flow and high pressures, and by transverse flow in and out of the fuel rod channels. An integral diffusion and vaporization test can be

TABLE 2
COMPARISON OF BOUNDARY LAYER CONDITIONS (CALCULATIONS AT 900°C)

Conditions	Symbol	FSV Reactor (Core Support)	Laboratory Conditions	
			HPTL	Low Flow Once Through
Regime		Turbulent	Turbulent	Laminar
Channel diameter, cm	d	17.8	1.0	
Pressure, MPa (atm)	P _t	4.7 (47)	4.7 (47)	0.1 (1)
Velocity, m/s	V	16.5	14	0.03
Reynolds number	Re	111,000	4922	0.17
Mass transport coefficient cm/s	k	3.0	5.3	0.82
Reaction rate/in-pore(a) diffusion coefficient, cm/s	k'	2.74	2.74	5.8
k/k'	Sh	1.1	1.93	0.14
Depletion at surface	%	47	34	87

(a)k' estimated for PGX graphite (log 138), which is highly reactive (1000 x H-451).

designed to measure these phenomena at reactor coolant conditions and be used to evaluate, modify, and verify the computer codes FIPER Q (Ref. 4), TRAMP (Ref. 5), and TRAFIC (Ref. 6).

Purification system studies are best performed at reactor conditions. Gas flow rate and pressure definitely affect mass-transport-controlled sorption and desorption in the low-temperature adsorber, the high-temperature adsorber, and the oxidizer bed. Such studies can be performed in the HPTL because two separate bypass purification trains have been provided, and these trains can be modified for any proposed experiments.

Plateout and liftoff of fission products such as iodine and cesium are controlled by the fluid dynamics and temperatures of the system in question. Integral tests can be performed in the HPTL using trace iodine or cesium, for example, to qualify or verify the plateout computer codes PAD (Ref. 7) or PADLOC (Ref. 8).

Although all of the codes mentioned have been used to predict chemical phenomena in reactors such as Fort St. Vrain, Peach Bottom, or Dragon, or in the in-site loop tests, it would be convenient to test the codes in a less complicated pilot system like the HPTL, in which test conditions and code input can be adjusted in parametric fashion. In this regard, the HPTL can be used to both develop and verify codes prior to using them in the highly complex reactor or in-pile loop situations.

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