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Precipitation and Scaling in Dynamic Geothermal Systems

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MASTER

OAK RIDGE NATIONAL LABORATORY

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PRECIPITATION AND SCALING IN DYNAMIC GEOTHERMAL SYSTEMS

E. G. Bohlmann, A. J. Shor and P. Berlinski

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SUMMARY

A dynamic loop facility for studying scaling in simulated geothermal brines - under conditions such as will be encountered in engineering scale heat transfer equipment is being constructed. The first phase of the program consists of two principal efforts:

1. Modification of an existing 100 gpm titanium loop to provide the engineering scaling test facility.
2. Operation of an ~1 gph once-through (experimental) system to provide design data for titanium loop modifications to provide experience with scale formation and characterization. This experience is being used in planning the scaling studies to be conducted in the dynamic loop facility.

INTRODUCTION

The ERDA Definition Report (ERDA 86) projects that significant utilization of geothermal energy through the year 2000 depends on three resources: Hydrothermal, Geopressured, and Hot Dry Rock. High temperature geothermal waters obtained from such resources will commonly contain appreciable quantities of dissolved silica which will become super-saturated on cooling with resultant precipitation and scaling propensities deleterious to the performance of heat transfer and mechanical equipment (valves, bearings, etc.). A perspective on the magnitude of the problem is supplied by estimates that the silica precipitation potential in various parts of a 100 MWe geothermal plant is approximately one ton per hour.

We have modified an existing 100-gpm titanium loop to provide a dynamic facility for studying the formation of silica precipitates,

their properties and fates, as a function of brine composition, temperature (to 300°C), and flow conditions. This loop demonstrated excellent serviceability over a period of years in saline water corrosion studies with and without pollutant additives such as H_2S , NH_3 , and SO_2 and should be equally satisfactory in this application.

The principal function of the modified loop will be to provide for circulation of silica saturated solutions of known composition through a heat exchanger-reheat bypass section in order to study silica deposition from the resultant supersaturated solution under conditions like those encountered in geothermal heat transfer equipment. Modifications of the bypass will also permit examination of surface and turbulence effects. Scaling in the cooldown section will be determined by following changes in heat transfer rates and physical examinations. Scales formed will be characterized by various techniques: metallography, crystallography, electron microscopy, and chemical removal.

Silica saturation of the circulating solution will be effected in another bypass section containing a saturator column loaded with powdered silica.

100 gpm Loop for Dynamic Scaling Studies. We have modified an existing 100 gpm titanium loop previously used for high temperature-high pressure corrosion studies. This apparatus was capable of circulating solutions at temperatures up to 320°C and 2000 psi using a 100 gpm (nominal) canned rotor centrifugal pump. The main loop piping consisted of an inlet and outlet manifold of 1-1/2 IPS titanium pipe, and an independently heated vessel referred to as a pressurizer held at a higher temperature than the loop mainstream for pressurization of the

circulating fluid. Heaters installed on the pump suction line and on the pressurizer together with the work done by the circulating pump provided sufficient makeup heat to maintain the system at the desired control temperature.

Loop Circulation System. A schematic diagram of the pump test loop is shown in Fig. 1 while Fig. 2 is a photograph of the system in its present location in the geothermal laboratory. The central component of the system, the circulating pump, consists of a modified Westinghouse Model 100 A canned-rotor centrifugal pump shown diagrammatically in Fig. 3. Depending on the size of the installed impeller, the pump was designed to deliver up to 100 gpm at a differential pressure of 100 psi while containing a system pressure up to 2000 psig. The squirrel cage induction type, 3-phase, 3400 rpm motor is totally enclosed (or "canned"). The motor rotor and bearings are bathed and lubricated by the liquid being pumped. The temperature of the fluid in the rear of the pump, as well as the temperature in the electrical stator windings of the canned motor stator, must be maintained between 50-100°C. The windings are cooled by circulating transformer oil and are isolated from the main circulating system by a .040 in.-thick metal diaphragm liner as shown in a sketch of the pump in Fig. 3. All materials wetted by the circulating solution are enclosed or constructed out of commercially pure titanium with exception of the bearings and journals which are fabricated out of a special dense grade of aluminum oxide. Labyrinth rings in the thermal spacer separate the pump impeller and volute from the motor section preventing extensive mixing of the hot solution into the cooler rotor and bearing region. In addition, seal

Fig. 1. Schematic diagram of 100A solution test loop.

PHOTO 0095-76

Fig. 2. Loop partly assembled in geothermal laboratory.

Fig. 3. 100A pump.

rings are installed around the front and rear hubs of the closed impeller to reduce pumping loss. The adjustment of these rings also serves in part together with the labyrinth rings in the spacer to control the axial thrust of the rotor which is thus designed to "float" and to minimize wear on bearing thrust surfaces.

The main flow of solution in the loop is from the discharge manifold through the various flanged bypass sections on to the suction manifold to the suction header containing the loop heater and thence into the pump suction. The mixing line takes off from the top of the inlet header and runs to the top of the pressurizer and returns from the bottom of the pressurizer into the circulating stream and from there to the pump suction. All the bypasses including the pressurizer mixing line may serve as locations for different experiments within the pressure-flow capacity of the pump. For example, these connectors may be fitted with experimental sections containing various material specimens and surfaces which are subjected to the controlled dynamic conditions of the circulating fluid for different time periods and environments. These experiment locations have been fitted with a silica saturator column, and a double tube cooler and reheater which are described below.

A high-pressure metering pump (Lapp Pulsafeeder Pump) is used to pump feed containing dissolved additives with the exception of silica at a rate of about 1 gallon per hour into the cooled rear of the pump to maintain the rotor bearings free of silica. Concurrently, a pneumatically operated valve controlled by a Foxboro pressure control system acts on a precooled and pretreated letdown stream and maintains

system pressure at the desired level, usually 1000 psig. In the process of cooling the letdown stream to approximately room temperature we will also study the cooler surfaces for silica scaling, however, under lower flow and less controllable conditions than in the loop mainstream.

Auxiliary equipment such as pumps, flowmeters, samplers, control and safety instrumentation, and analytical equipment are in place to complete the experimental system. The high pressure, high temperature loop components are thermally insulated and contained in a 1/8" thick steel enclosure. This space is exhausted via a blower and dust system to the outdoors. Heater and pipe temperatures throughout the loop systems are recorded, critical temperatures being interlocked with power panels to control and shut down loop operation in the event of excessive temperature. Excessively high or low pressure, the latter of which could damage the circulating pump and loop from cavitation and overheating, are similarly interlocked with the pressurizing feed pump and heater power supplies.

Feed Preparation and Injection. To prepare a simulated geothermal brine various salts are dissolved in a weighed makeup tank. The solution is transferred to a large enclosed 400 gallon stainless steel tank equipped with stirrer and sparging where appropriate gases may be added and overall concentrations adjusted prior to feeding to the loop. The feed solution at room temperature is injected with a Lapp Pulsafeeder Pump into the back of the circulating pump at a rate of about 1 gallon per hour, helping to flush and maintain the bearings in 50-100°C operating range and preventing backup of loop solution and possible silica precipitation in this region. (This purge rate has

been shown experimentally to be adequate to protect the pump bearings when slurries containing 1000 g/liter thorium oxide were being circulated by pumps of this design.)

Silica Saturation. To simulate various geothermal brines containing silica it is necessary to maintain a controlled level of dissolved silica in the circulating solution. Part of the loop flow is bypassed through a silica column at a nominal rate of 1 gallon per minute where it equilibrates with the solid amorphous silica powder in the column to a concentration corresponding to the loop and column operating temperature. (Exploratory studies with a smaller column have shown reasonable agreement with literature data on amorphous silica solubility in water achieved with a single pass through a similar column.) The silica saturator is piped between flanges in the inlet and outlet manifolds of the loop as shown in Fig. 4. An orifice meter in the outlet flange is used to monitor flow through the saturator while a manual valve is used to isolate and control flow through the column. At a solution flow rate of 1 gpm, a feed rate of 1 gph and a loop volume of ~6 gallons, saturation concentrations of silica are reached in less than an hour following the start of the experiment.

The silica saturator consists of a section of four inch IPS schedule 80 (3.826 inch I.D.) pipe, 24 inches long, welded to a conical discharge section which contains a porous tantalum 70 μ m pore size support frit. The top of the saturator is fitted with a 4 inch-900 lb flange and mated with a flange to which is connected a 1/2 inch solution inlet line and 1-1/4 inch silica loading port. Column loading is approximately 3.6 kg of silica (-140 + 200 mesh, 75-100 μ m) for a 4.5 l

Fig. 4. Saturator column.

column volume and density of 0.8 g/cm^3 . Based on a saturator solution concentration of 1000 ppm, corresponding to a 225°C operating temperature with amorphous silica, and a 75% bed depletion approximately 700 hours of operation may be feasible before renewal of silica will be necessary.

Letdown System. Solution saturated with SiO_2 at the loop temperature, pressure, and dissolved solids concentration enters the letdown system from the top of the pressurizer at a flow rate of about 60 ml/min (1 gal per hour). This stream, particularly as noted in work with a small single-pass hood system, is useful for the study of silica deposition under near static conditions at various temperature ranges. By flowing through a double pipe heat exchanger and controlling the temperature gradient of the process fluid using various coolant flow rates through the annulus between the inner and outer pipe, scale is deposited under controlled conditions. The inner (process) line in the heat exchanger may be constructed of tubing of different materials of interest, and inspected for scaling following the run at various points of the temperature gradient. Provision is made for injection of a concentrated acid or base side-stream ahead of the control valve to control deposition of scale on the valve and which will upset loop pressure control. Various devices such as orifices or venturis may be installed to maintain a desired pressure gradient at temperatures of interest to study the effect of a sudden phase change or flashing on deposition of scale. Figure 5 is a schematic of the double tube heat exchanger letdown system.

Fig. 5. Schematic of letdown heat exchanger.

Sampling. Grab samples of ~ 30 ml volume are regularly removed from the loop. In order to prevent flashing, possible solids deposition, and composition variations, we have designed our sampling to be performed under isothermal and isopiestic conditions during sample removal. Figure 6 shows an exploded drawing of the sampler. It is constructed of a stainless steel chamber containing an enclosed piston. The rear of the piston is backed up by an adjustable liquid pressurization system maintained equal to or slightly less than the loop pressure. Blowby between the front section of the chamber attached to the loop and the pressurized liquid rear section is prevented by several Viton "O" rings on the piston. Following purging of the sampling tee valves, the sample cylinder is attached to the sample tee and liquid pressure behind the piston is slowly lowered permitting loop sample liquid to enter and fill the available volume of the cylinder, the latter having been insulated and preheated to the loop operating temperature.

Bypass Heat Exchanger-Cooler. The bypass heat exchanger shown in Fig. 7 and Fig. 8 is intended to provide realistic flow conditions, i.e., linear flow rates of 4-6 feet per second, Reynolds' numbers ranging from 10^4 to 10^5 , depending on conditions, for the study of the effects of silica scaling on heat transfer rates at various geothermal brine temperatures and environments. The brine flows at a nominal rate of one gallon per minute through sections of 1/4 inch schedule 80 titanium pipe. Cooling water flows in the annulus of an 1 inch nominal ID copper tubular jacket surrounding each cooler section. The annuli are sealed with swaged type heat exchanger tees. Depending on the range of cooldown temperatures and the temperature distribution desired,

DIMENSIONS IN INCHES

Fig. 6. Silica solution sampler.

ORNL-DWG 76-13311

Fig. 7. Bypass heat exchanger for scaling tests.

ORNL-DWG 76-13312

Fig. 8. Bypass heat exchanger detail.

up to 5 cooler sections each with an effective length of ~ 7 feet may be installed in series permitting the brine temperature to drop, for example, from 250°C to 100°C in five temperature increments. Figure 8 is a diagrammatic sketch of the arrangement of coolers. Every cooler is identical, permitting ready substitution; each may be inspected in situ by removing blind end-flanges and inserting a boroscope into the pipe. Specimen of scale may be removed. Well type thermocouples monitor inlet and outlet temperatures of both process fluid and coolant for each double tube section. Heat transfer from the process fluid through the inside liquid film, the deposited scale, the titanium pipe wall and into the coolant water may be estimated. By computing both inside and outside liquid film heat transfer coefficients from standard liquid film correlations and correcting for the conductivity of the intermediate titanium pipe wall, the contribution of deposits may be estimated and compared to "clean" running conditions. Thus, scaling may be pinpointed throughout the brine temperature gradient. Specimens of various shapes and materials may be mounted in connecting sections joining the double pipe heat exchangers at selected isothermal positions to study effects of impingement, wall shear stress, turbulence as well as material composition and structure on scale deposition. The connecting sections are also useful for corrosion testing of materials at a variety of temperatures and flow conditions. Corrosion specimens may also be mounted in other bypass sections in the loop, albeit at the operating mainstream temperature. Process liquid flow through the cooldown bypass is monitored with a calibrated venturi in the inlet (highest temperature section). A high temperature titanium manual-control

valve fitted with a lock is also located in the inlet section of the cooldown array to isolate and establish desired flow rates through the bypass.

To guard against boiling in the inlet coolant section, the area of highest process temperature, cooling water pressure will be raised to over 200 psi ($\sim 200^{\circ}\text{C}$) with a multiple stage centrifugal pump. At a coolant flow rate up to 5 gpm subcooling and wall temperatures anticipated in the annulus are such that no phase change should occur in the annular shell.

Bypass Heat Exchanger-Reheater. The 1 gpm bypass stream after being cooled to $75\text{--}100^{\circ}\text{C}$ is returned to the main loop at the top of the pressurizer via a set of series connected heaters (see Figs. 9 and 10). The five $1/4$ " schedule 80 titanium pipe sections are enclosed with tubular heating elements which collectively add ~ 40 kw to the 1 gpm bypass stream. Starting with fluid at a mainstream temperature of 280°C this power input represents about 90% power return to the bypass stream. The remainder is supplied by the main loop heaters and by pump work. Each heater subassembly consists of 4 Incoloy-sheathed tubular heating elements $1/4$ " in diameter, 8 foot long, each with 2 kw capacity, symmetrically spaced over the outside of the $1/4$ " pipe ($.540$ inch outside diameter) and secured to the pipe at intervals with stainless steel clamps. To improve thermal contact between adjacent heaters and pipe wall, Thermon type T-63 heat transfer cement is applied to fill spaces between the heaters and to add a $1/2$ " thick layer over the elements. Each heater subassembly is insulated with 2" of Fiberfrax ceramic fiber insulation. Room temperature tests and computational

Fig. 9. Bypass Calrod reheat system detail.

Fig. 10. Bypass Calrod reheat system.

approximations indicate that the maximum sheath temperature at the highest process temperature location (at the outlet of the reheat section) will not exceed the maximum allowable Incoloy sheath temperature of 1500°F but will be of the order of 1000°F, at the rated power input. Stresses set up by thermal and mechanical gradients at a power input of 1 kw/ft at 1000 psi at highest temperature operating point are not sufficient to exceed the allowable stress limitations of the titanium pipe by at least a factor of 2.

Temperature, Pressure, Power Monitoring and Safety Interlocks.

Process and coolant temperatures associated with the double pipe cool-down sections are carefully monitored to follow heat transfer changes closely and accurately. In addition thermocouples are located at many points around the loop for monitoring loop and pressurizer heating elements and adjacent process temperatures. Circulating pump stator temperature and power are also monitored.

A SCR temperature control system with proportional band and reset action will supplement the on/off loop temperature control system. This will operate on the final 8 kw power input required in the reheat section of the heat exchanger bypass. In the absence of a heat exchanger bypass, on/off control is adequate. Remaining heaters in the bypass are manually controlled in groups of 2 kw. Operating pressure for readout and control is recorded on Foxboro recorder-controllers while differential pressures across orifices and the silica saturator column are transformed via Foxboro pneumatic transmitters to standard 3-15 psig signals and recorded. Load currents from the 100A pump and various heaters are monitored and all current demand devices have

overload protection either through high current or high temperature power controls. To protect the circulating pump, the pump and all heaters are shut down by redundant low pressure switches. Loop integrity is protected by a high pressure feed pump power cutout as well as with a pneumatically controlled valve on the letdown system. Many of these safety functions will acquire additional redundancy upon the installation of a data processing system similar to the Hewlett Packard 3050B.

Data Processing and Analysis. In our investigation of scaling, the several sections of a double-pipe heat exchanger are individually monitored for process and coolant inlet and outlet temperatures, pressures and flow. These data together with the physical-chemical properties of the fluids and materials such as viscosity, density, heat capacity, thermal conductivity all as a function of temperature and pressure are considered in computing apparent process side and coolant side heat transfer coefficients, as well as changes in the heat-transfer coefficient as a function of process time at various geothermal brine compositions.

An on-line programmable computer-based data acquisition system is necessary in conjunction with extended experiments. In many cases runs lasting several weeks will be required to develop and follow silica scale formation. A machine capable of logging data hourly or shorter intervals, performing heat transfer computations and storing results on tape for future processing while simultaneously performing control and safety functions will save considerable operating and computation time. Data may be summarized, tabulated, graphed and studied

at the experimenter's convenience.

A request for capital funds has been written and accepted for an in-line programmable computer for data storage, analysis, and processing. In addition, a proposal has been written to the Automatic Data Processing Committee of the Oak Ridge National Laboratory which studies and disposes of such requests. Finally, specifications were written preparatory to issuance of a requisition requesting bids from various suppliers.

Specifications include a general purpose digital controller interfaced with a 40 channel scanner, analog-to-digital multimeter converter, digital X-Y plotter shown in block diagram in Fig. 11. The system will initially have the capability of accepting up to 30 channels of guarded, low thermal EMF, differential DC voltage inputs and will include 10 channels of DPDT relay contact outputs.

The general-purpose stored-program digital controller with 11.6 K words of memory will control all data acquisition, generate output signals for experiment control, convert raw data to engineering units, perform programmed calculations and manipulations on the data, and output the processed data to magnetic tape cassette for storage or (in the form of tabulations or graphs) to a strip printer or X-Y plotter. The controller will be programmed in a high-level language and system programs will be storable on magnetic tape cassettes. The controller features a live keyboard making possible on-line examination and modification of program variables and constants; it will be possible to disable the keyboard to protect the system during unattended operation.

The system will be readily capable of expansion by the addition

Fig. 11. Block diagram - geothermal brine precipitation and scaling data acquisition system.

of a second input-output scanner permitting up to 40 additional channels. Up to a maximum of 15 peripherals or accessories may be interconnected using the proposed system.

ONCE-THROUGH DEVELOPMENT SYSTEM

Initial consideration of the design of the dynamic, heat exchanger bypass 100 gpm loop system demonstrated the need for a development system for defining design parameters. The system was initially conceived as supplying pressure drop and equilibration rate data for the design of the saturator column, but it so rapidly demonstrated more general usefulness that it has been upgraded to the system shown in Fig. 12 and continues to evolve as we learn from it. In all cases the system has consisted of a feed tank, high pressure diaphragm pump, preheater, saturator column and letdown system. Earlier systems were generally simpler and designed for daytime operation only, but sufficiently like the present system that we shall forego detailed descriptions at this point. Presently the system is instrumented and otherwise equipped to operate overnight unattended, but equipment scaling problems have, thus far, interrupted such operation.

Description of Present System.

Feed tank - 30 gal. polypropylene tank, bottom outlet, replenished from 150 gal. makeup tank as required.

Feed pump - variable (to ~1 gph) pulsafeeder pump with stainless working head which will deliver to ~2000 psi.

Preheater - "Kanigen" nickel plated 3/8" carbon steel pipe traced with a 2 kw "Calrod" imbedded in Thermon and suitably insulated.

Saturator column - 1" dia. 316 SS column 15 inches long with

Fig. 12. Once-through development system.

tantalum frit to support the charge of amorphous silica. Wrapped with a 1.5 kw Calrod heater imbedded in Thermon and suitably insulated.

Intermediate oil bath cooler - 3/8" carbon steel pipe in silicone oil, thermostatted using Calrod heater and cooling coil.

Letdown system - assemblage of valves, samplers, and glass tubing or pipe for observing scaling. The valves are usually air operated control valves used to adjust pressures to those desired in the various parts of the system: prevent boiling in the saturator column, reduce pressure to a tolerable level in the glass components.

All components are instrumented to provide desired temperature control.

STUDIES WITH ONCE-THROUGH SYSTEM

Amorphous Silica for Loading the Saturator Column. The first use of the once-through system was the identification of an amorphous silica material suitable for loading the saturator column. A promising candidate material suggested by the work of Elmer and Nordberg¹ was porous Vycor (Corning Glass Code #7930, 97% SiO_2 , 3% B_2O_3) which could be obtained as a specified mesh size powder from Corning Glass Company.

Exploratory studies with some of this material (-325 mesh) in a 1 cm diameter column at temperatures to 250°C gave reasonable agreement with literature data for amorphous silica solubility in water, as shown in Fig. 13. Some of the data at ~240°C were obtained with only about a third of the silica left in the column indicating saturation was achieved with a contact time of < 12 sec. The pressure drop through the column, however, was excessive - the pressure available from the 100 gpm pump is about 100 psi. Calculations²

Fig. 13. SiO₂ solubility.

indicated that a -140, +200 mesh powder would be suitable so additional Vycor powder in that size range was obtained. Subsequent tests in a 1" diameter column substantiated this as did initial tests with the 4" diameter x 28" saturator column in the 100 gpm loop. With the column ~60% full the pressure drop observed was ~15 psi compared with a calculated value of 22 psi for the column when full.

Stock Solution Preparation. In order to compare the behavior of the supersaturated silica solutions prepared with the Vycor column with similar solutions prepared by other techniques we initially used the approach of varying the composition of the feed to the column to produce an effluent of desired composition. This approach, however, soon proved unwieldy because of system holdups, column interactions with the feed solutions, and scaling problems in the letdown system (see later section), so we changed to preparing stabilized monomer stock solutions for laboratory experiments. Cordurier and co-workers³ and others showed that the rate of polymerization of monomer is extremely pH dependent in the range 1-9 with the minimum rate occurring at pH 3 in dilute solutions, see Fig. 14. We have briefly examined the effect of pH on the rate of polymerization of monomer prepared with our once-through system with results consistent with those of Cordurier, et al. as shown in Fig. 15. The substantial effect of pH on the rate of monomer polymerization is further emphasized by the data in Fig. 16. These data show the effect of various salts (all at ionic strength = 1) on silica monomer polymerization. Similar to the effects observed in colloid precipitation, the calcium chloride increased the rate much more than the sodium chloride; the trivalent

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Fig. 14. Effect of pH on the initial rate of silica monomer polymerization.

Fig. 15. Rate of monomer-polymerization vs pH.

Fig. 16. Effect of salts on silica monomer disappearance.

aluminum salt, however, was completely ineffective at the lower pH of 2.9 required to avoid hydrolysis. This suggested that pH had the stronger influence, which was confirmed by repeating the calcium chloride experiment at pH 2.9 with resultant stabilization of the monomer in the calcium chloride solution.

Scaling Studies. Scaling studies were initiated in the once-through system to develop a background of experience on the effects of solution composition on scale formation, to provide scale samples for development of scale characterization methods, and to develop methods of keeping letdown valves operable. Rapid plugging or seizure of these valves has been the most frequent cause of interruption or termination of operation of the once-through system; this will be discussed in the following section.

During the operation of the once-through system for evaluation of the porous Vycor for use in loading the column and for preparation of supersaturated silica solutions for the other studies described, no evidences of scale formation were encountered. In most of these studies the feed was distilled water which produced an effluent from the system with a pH of 4.5 to 5. Since silica scale formation is generally considered to be related to monomer polymerization, we decided to accelerate that reaction by raising the pH to see if we could produce conditions under which the solutions prepared from the porous Vycor would indeed produce scale. Subsequent operation of the system gradually increasing the pH of the feed solution with NaOH additions soon produced scale in the quartz tube cooldown system as shown in Fig. 17. Analyses showed the deposit to be a varied mixture of silica and iron

PHOTO 3324-75

Fig. 17. Scale formed - quartz cooldown sections.

oxide, (Fe_2O_3 , Fe_3O_4) corrosion products from carbon steel parts of the system. These operations also demonstrated an anticipated problem: rapid complete plugging of the letdown valve (see below).

Having demonstrated that scaling would occur in the quartz tube cooldown system we decided to go to a Pyrex glass pipe system to make it possible to go to higher pressures, and thereby temperatures, than the 25-50 psi possible with the quartz tubing and O-ring fittings used in the previous experiments. We preferred to stay with glass so we could see the patterns and locations of scale formation in the continuing exploratory studies. We have found that with careful assembly using the standard gaskets and flanges, we can operate to 175 psi and a temperature range from 115° to 35°C in the glass pipe (3/8") system shown in Fig. 18. The figure shows the present version of this system, which has been undergoing considerable evolution as we experiment with it. It is made up of a series of 6" (c) and 36" (A-A) straight and 6" bent pieces of 3/8" Pyrex pipe assembled as shown. The supersaturated silica solution from the oil bath cooler enters at -100°C at the lower right, and cools to 35 to 50°C, depending on flow rate ($35-75 \text{ cm}^3/\text{min}$), as it traverses the glass system. We use the several segments of the system as follows:

The 6" pipes are tared before assembly and then dried and weighed after a run to get information on amounts of scale deposited as a function of temperature. We will also attempt to get information on hydration of the scale by weighing after drying at several temperatures; weighing errors and other interferences will probably limit this possibility to rather heavy deposits, however. After the weight data

Fig. 18. Pyrex pipe cooldown test section.

have been obtained from these sections, they will be broken to provide in situ specimens for physical examinations: e.g., optical and electron microscopy and diffraction analysis.

The 36" sections will contain metal alloy specimens at various points (A and B for example) to provide information on substrate effects. The shape of these metal specimens is shown in the upper left hand corner of Fig. 18. The tabs at each end of a specimen are bent to "anchor" the specimen in place in the Pyrex pipe. The narrow flat sections can then be inserted, removed, and examined without physical alteration with consequent possible effects (cracking, spalling) on the deposits; the flat sections can also be oriented horizontally, vertically, etc. in the pipe. The 36" sections are also available for testing physical and chemical methods of removal. Preliminary results indicate substantial variation of properties depending on the temperature at which deposition took place. Those formed at $\sim 100^{\circ}\text{C}$ are very hard and adherent and it has taken 20-30 hrs of treatment with 50% sodium hydroxide at 70°C to substantially dissolve them from the Pyrex pipe surface; they can be scraped off only partially and with considerable effort. As the temperature of deposition drops, removal by mechanical means or using the NaOH treatment becomes progressively easier. At 40 to 50°C most of the deposit can be removed with a test tube brush and the 70°C sodium hydroxide treatment is effective in 20-30 minutes.

We have just begun the scanning electron microscope examinations of the in situ deposits after drying.

Letdown Valve Plugging. In the first versions of the Once-Through Development System the letdown valve was placed just downstream of the

oil bath cooler. This location was chosen to assure a tolerable pressure in the glass cooldown test section. The oil bath cooler reduced the temperature to a point such that flashing would not occur across the valve with even greater likelihood of scaling. Also, the experience at Cerro Prieto where little or no scaling occurred in the heat exchangers with effluent temperatures at $\sim 135^{\circ}\text{C}$, suggested that limiting the degree of supersaturation by letting down at the higher temperature (with consequent higher silica solubility) might eliminate or minimize scaling in the valve. Unfortunately, this did not prove to be the case. Plugging of the valve occurred whenever conditions were present which produced scale in the glass cooldown system, i.e., effluent pH greater than ~ 6 in water or 0.1 M sodium acetate feeds. Typical deposits are shown in Figs. 19 and 20.

We have tried various valve trims such as stainless steel, titanium, and gold-plated and Teflon-coated stainless steel without observing appreciable differences. Injection of sodium hydroxide (to produce 1 N in mixture) just before the letdown valve rapidly clears up incipient plugging, but, of course, also makes the solution non-scaling in the cooldown system.

Placement of the letdown valve downstream from the Pyrex cooldown system as shown in Fig. 18 resulted in operation without difficulty continuously for 43.5 hrs under mildly scaling conditions (pH 6.15, 0.1 M sodium acetate feed, 850 ppm SiO_2). Injection of base or acid (pH 3 in mixture) would permit this mode of operation under more aggressively scaling conditions. However, this approach limits the temperature at which the entire system can be operated to pressures

PHOTO Y-135522

Fig. 19. Scaled SS valve stem.

PHOTO Y-135596

Fig. 20. Scaled valve seat.

tolerable in the Pyrex cooldown system or $\sim 175^{\circ}\text{C}$ in the saturator column.

On the basis of the observations made, we believe we can operate with the letdown valve just downstream of the oil bath if we have the pH about 4-4.5; we will then inject a concentrated buffer solution downstream from the valve to produce the desired pH in the Pyrex cooldown system.

PROGRAM

A program plan outlining anticipated activities during the rest of the calendar year is attached. During the next quarter effort will be concentrated on completion of the 100 gpm Dynamic Scaling System.

PROGRAM PLAN: PRECIPITATION AND SCALING IN DYNAMIC GEOTHERMAL SYSTEMS (EB 01 02 02 0)

System and Major Tasks	May	June	July	August	September	October	November	December	January	February	March	April	May	June	July	August	September
Once-Through Development System	Assemble modified system for operation	Prepare monomer stock solutions for bench scale experiments and study effect of brine concentration on scaling in Pyrex let-down system				Study effect of monomer concentration and temperature on scaling as time permits				Prepare report on once-through experiments				Conduct exploratory studies on effects of heavy metals and sulfides as time permits. (Emphasis will be on studies with heat exchanger loop system)			
100 gpm Heat Exchanger Loop Saturation Column checkout	Prepare SiO ₂ and load saturator column	Test saturator column and let-down system performance. Use acid or base injection to prevent letdown control valve plugging												Conduct studies of effect of pH on scaling in heat exchanger: 100-200 h runs; pH 5-8, 0.1 N sodium acetate, ~ 20,000 ppm chlorides, ~ 900 ppm SiO ₂	Effect of brine concentration: same as earlier runs except pH 6.1 and brine 2000 - 200,000 ppm chlorides	Effect of supersaturation concentration	
Heat Exchange bypass for dynamic scaling studies	Construct segmented titanium cool and reheat system and associated connectors, mounting rack, and containment	Assemble and connect to 100 gpm loop system	Install cooling water system	Check and adjust flow rates and instrumentation	Make necessary modifications and insulate	Carry out manual shake-down run with scale-forming conditions from once-through experience											
		Install instrument panel and mount instruments		Install and connect sensors, etc.													
			Design and construct permanent letdown system		Install let-down system												
Data logger and processor	ORNL approvals	Out for bids	Place order	-----Twelve weeks delivery-----			Assemble system and check out	Prepare software and interface computer and loop system	Debug system with water in loop					Completion Report on 100 gpm			

Study Reactions of Silica Species with Solid Amorphous Silica	Build bench scale apparatus	Investigate effects of pH, monomer and polymer concentrations, temperature, brine concentration (summer student)	Write report
Status Report		Final draft to reproduction	

*Asterisk indicates estimated beginnings and ends of studies described by the legends.

FY-77 Cost Breakdown (10 ³ \$)		
	Once Through System	Heat Exchanger System
Staff	36	140
Support Labor	10	44
Materials	4	20
Analytical Support	12	8
Miscellaneous	5	21
Total	67	233
Transition	20	55

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