MECHANICAL AND TRANSPORT PROPERTIES OF ROCKS AT HIGH
TEMPERATURES AND PRESSURES

TASK II

FRACTURE PERMEABILITY OF CRYSTALLINE ROCKS AS A FUNCTION OF
TEMPERATURE, PRESSURE, AND HYDROTHERMAL ALTERATION

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PART I

EXPERIMENT AND EQUIPMENT DESIGN CONSIDERATIONS FOR MEASUREMENT OF PERMEABILITY VARIATIONS ASSOCIATED WITH FLUID/ROCK CHEMICAL INTERACTIONS AT ELEVATED STRESS AND TEMPERATURE STATES

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ABSTRACT

Pore-fluid chemical interactions on both short and long time scales can significantly change the permeability of a rock. Measurement of the permeability variations requires adaption and modification of standard measurement systems with special attention given to pore-fluid flow rates and metal corrosion of system components. In this report, system requirements and capabilities are reviewed, analyzed and recommendations made. Special attention is given to the choice of corrosion resistant metals, fluid-flow systems, back-pressure systems, jacketing materials and flow-rate measurement. On the basis of this study, an economical, highly flexible, permeability system was designed and built. The system allows measurement of permeability over the darcy to nanodarcy range using geologically meaningful, chemically reactive, pore fluids under constant volume flow rates as small as 0.2 ml/day at temperatures in excess of 300°C, fluid pressures to 20 MPa and confining pressures to 100 MPa.

A brief overview of the system follows. Fluid flow is generated by an upstream servo-controlled positive-displacement syringe pump that can be operated to generate either a constant prescribed flow rate or a constant differential pore-pressure across the sample. Under constant flow-rate control, the system can achieve flow rates as low as 0.2 ml/day and as high as 4800 ml/day at fluid pressures to 20 MPa. Back-pressure can be regulated by either a second, downstream servo-controlled syringe pump or an adjustable spring-loaded pressure relief valve. All wetted components (tubing, valves, tees, reservoir, specimen end pieces) subjected to elevated temperatures or in contact with corrosive pore fluids are made of the Ni-Cr-Mo alloy, Hastelloy C-276 (11,000 psi limit). All other tubing and components are

316 SS (11,000 psi pressure rating). A 55 ml, 30 cm long, tubular reactor made of Hastelloy C-276 serves as an upstream reservoir for corrosive fluids at elevated temperatures. A 2 micron Hastelloy C-276, in-line filter is positioned immediately upstream of the sample outside the pressure vessel. The sample, jacketed in either teflon or gold (depending upon the temperature) is hydrostatically loaded (up to 100 MPa) in an externally heated, high-pressure, Aminco hydrothermal reaction vessel modified to allow through flow of the pore fluid. The pressure drop across the sample is measured using a high resolution, variable range, differential pressure transducer capable of measuring a ΔP as small as 1 Pa to as large as 20 MPa (0.0001 to 2900 psi). Flow rate is measured using a flow gage constructed from high pressure capillary tubing and another high resolution differential pressure transducer. The system has a microcomputer for data acquisition and servo-control of the pumping rates of both the up- and downstream pumps. Permeability can be measured in either of two modes: (1) continuous, constant flow for high to medium permeabilities, or (2) pressure-pulse decay for permeabilities in the microdarcy to nanodarcy range.

INTRODUCTION

The initial long-term objective of the research was to measure and understand the variation of fracture permeability in several crystalline rocks (specifically: Sioux Quartzite and Westerly Granite) as a function of hydrothermal reaction with reactive through-flowing pore fluids (H_2O and aqueous Na_2CO_3 and $NaCl/MgCl_2$ solutions) at temperatures to $300^{\circ}C$, fluid pressures to 20 MPa and effective pressures to 70 MPa. Experiments were planned to evaluate the relative importance of dissolution and secondary mineral formation. Emphasis ultimately focused on the effects of dissolution.

Critical to the proposed research was the design and construction of a permeability system that would be compatible with highly corrosive fluids at elevated temperatures and pressures. At the initiation of this project, however, no proven permeability systems were known to be in existence that could meet the requirements necessitated by the research objectives and physico-chemical conditions associated with the planned experiments, especially as related to metal-corrosion resistance and flow-rate control requirements. This is because the preponderance of permeability research up to that time had dealt with only the physical effects of the stress state upon permeability. There had been few specific studies of the chemical effects associated with fluid/rock interaction upon permeability variations. These earlier studies utilized distilled water or low salinity water as the working fluid and the assumption was made (sometimes falsely) that fluid/metal reactions were small and insignificant. The use of corrosive fluids requires all wetted parts of the permeability system, especially those components at elevated temperature, be resistant to corrosion with respect to the working fluid. Unfortunately, there is no single readily available, economical metal that serves as a panacea for all pore-fluid compositions and temperatures.

In addition to system modifications to minimize corrosion effects, the system must be designed so that the pore-fluid flow rate can be controlled. In the planned experiments, pore-fluid flow rate is an important parameter to be controlled because it influences mineral alteration/dissolution/crystallization kinetics as well as induces migration of very fine-grained particulate and colloidal material.

For example, pore-fluid flow rate affects the kinetics of dissolution. If the dissolution is a transport-controlled reaction such as for calcite at $pH < 4$ (Solberg and Rickard, 1984), then the rate of dissolution is a strong function of the flow rate adjacent the surface. Flow rate is important even if dissolution is surface-reaction controlled, such as for calcite at high pH or quartz. In this case, the degree of undersaturation of the flowing fluid with respect to the reactive solid affects the dissolution rate. The degree of undersaturation changes, however, through time. If the rate of fluid flow is slow relative to the rate of dissolution (i.e. a long relative residence time in the sample), then the degree of undersaturation of the fluid decreases significantly along the flow path and dissolution effects will vary significantly along the length of the sample. In contrast, if the fluid flow rate is high relative to the rate of dissolution (short relative residence time), then the degree of undersaturation will vary only slightly along the length of the sample and dissolution modifications will exhibit less spatial variability.

The development of a spatial variability along the sample must be considered when establishing the experimental parameters to be used. If the primary objective is to measure the relationship between dissolution modifications and variations in fracture permeability, then it is most desirable that the dissolution effects be approximately uniform along the length of the fracture so that the measured permeability is representative of the fracture as a whole and not a weighted average. The fluid-flow rate must be controllable to assure this requirement. In contrast, if one desires to study the evolution of dissolution modifications, then flow rates must be sufficiently slow that significant spatial variability is developed. In this case, the measured permeability represents a weighted average of the permeability of the fracture.

Experiment design requires the permeability system possess the following capabilities:

- 1) temperature capability to 300°C as a minimum;
- 2) hydrostatic loading of samples to confining pressures to 100 MPa at 300°C;
- 3) compatibility with highly corrosive fluids at elevated temperatures, particularly Cl-rich solutions;
- 4) capable of measuring samples with a wide range of permeabilities: 10^1 – 10^{-8} darcy;
- 5) capable of measuring permeabilities using only a small differential pore-fluid pressure acting across the sample;
- 6) capable of controlled, long-term flow (at least 2 weeks); and
- 7) a wide range of controllable, constant pore-fluid flow rates, with special attention to allowing very low flow rates (< 1 ml/day).

The following topics and subsystems were most critical in finalizing the ultimate design and are discussed separately subsequently:

- 1) choice of corrosion-resistant metal for pressure supporting components at elevated temperatures;
- 2) flow-rate control system;
- 3) back-pressure control system;
- 4) permeability measurement system allowing both continuous flow and transient pulse methods;
- 5) jacketing materials compatible with both corrosive fluids and high temperatures;
- 6) automated control and data acquisition system for long-term flow experiments;
- 7) heater design and temperature control, including assurance of temperature uniformity; and
- 8) flow-rate measurement system.

GENERAL CONSIDERATIONS AND DESIGN DECISIONS

Corrosion-Resistant Metals

Choice of metals for minimization of corrosion depends upon several technical and practical factors, including anticipated pore-fluid compositions and temperature. Three high corrosion-resistant metals have received most extensive use for pressure supporting components: High purity Titanium, Hastelloy C-276 and Inconel 625; the latter two are Ni-alloys with significant Cr and Mo and have similar performances. Tubing, valves, fittings and reaction vessels are available but costly, although increased supply/demand has resulted in some reduction of prices. No one metal is a panacea for all pore-fluid/temperature conditions. For example, the two Ni-alloys

exhibit excellent resistance to hot aqueous Na/Mg chloride solutions but are less resistant to hot aqueous HCl solutions because of their Cr content. In this case, a Ni-Mo alloy (e.g. Hastelloy B) performs significantly better. The Ni-alloys perform well in both oxidizing and reducing environments, whereas Ti is best in oxidizing environments. Use of stainless steels for components in contact with corrosive pore fluids should be avoided if permeability measurements are to be made, as demonstrated by the results of Potter et al (1981) and Stottlemire et al (1981). Gold-plating of stainless steel components has been used by some workers but problems have been reported related to small pits in the plating or development of small blisters under the gold plating.

Ultimately, the decision was made to construct the system from a combination of Hastelloy C-276 and 316 SS. The 316 SS is used only for those parts of the system at room temperature and not in contact with corrosive fluid prior to entering the sample. A 55 ml tubular reaction vessel made of Hastelloy C-276 is located upstream of the sample and serves as an in-line reservoir. The working fluid is displaced from this reservoir by distilled water delivered from the upstream pump.

Flow-Rate Control Systems

Continuous flow can be achieved in rock samples with permeabilities greater than 0.1 to 1.0 microdarcy, although thermal effects associated with thermal expansion of pore fluids introduce difficulties at the lower flow rates. Continuous flow of fluid at pressure can be achieved by several means but only two systems allow pulseless control of flow rate.

Servo-controlled, positive-displacement syringe pumps provide the best means of obtaining controlled flow rates. Currently, relatively inexpensive commercial models are available with controllable flow rates to as low as 0.03 ml/day at fluid pressures to 69 MPa. At the time of construction of our system, these pumps had an upper pressure range of 20 MPa and a minimum flow rate of 0.2 ml/day.

Continuous controlled flow also can be achieved by use of two gas-backed, servo-controlled measuring separators or intensifiers, but are much more restrictive in their ability of prescribed controlled flow rates, especially at the lower flow rates. The limiting factor is the control of the gas pressure, which is limited to a value of + 5 psi. Two gas-backed separators are required, one upstream and one downstream of the sample. The pressure difference between the two separators is adjusted to achieve the desired flow rate as determined by measurement of the rate of stroke. One separator is kept at a constant pressure and establishes the back pressure, whereas the other is adjusted to achieve the prescribed flow rate.

A syringe pump is used in our system because it appears to offer greater ease of use and finer control of flow rates, especially low flow rates in the high permeability range typical of fractures subjected to low effective normal stresses.

Back-Pressure Control Systems

Pore-fluid back-pressure can be controlled to varying degrees of sensitivity by use in the downstream region of: (1) one or more micrometering valves in series, (2) adjustable "constant pressure" relief valves, such as a gas-backed dome regulator

or a spring-loaded relief valve, (3) a gas-backed separator with a high precision adjustable pressure regulator, and (4) a servo-controlled positive-displacement syringe pump.

Use of micrometering valves requires use of an upstream filter to capture suspended particulate material that can plug and change the flow characteristics of the valve, especially for cases of low flow rates at high fluid pressures. Micrometering valves are more appropriate for high flow rate situations, but several micrometering valves are available that have sufficiently small flow coefficients that moderately low flow rates can be obtained for water at pressures up to about 10 MPa. Based on our experience, it is recommended that micrometering valves be used only as a last resort, especially for long duration, unattended flow of fluids with the possibility of colloidal particles.

We have no experience with gas-backed dome regulators but have found that adjustable spring-loaded pressure relief valves provide an inexpensive means of obtaining a sensitive control of back pressure with only small dynamic pressure fluctuations. When using a spring-loaded pressure relief valve, attention must be given to the "stiffness" of the component in order to minimize the pressure fluctuation associated with the opening and closing of the valve. We and other workers (Gobran et al, 1981) have observed that these valves have increased leakage with time as the seating characteristics change. Frequent cleaning is recommended.

We have not used a regulated gas-backed separator for back-pressure control. The limiting factors of this type of system are the sensitivity of the pressure regulator and the friction of the pressure seals on the moving piston. Other workers (Terra Tek Inc., personal communication) report that back-pressure regulation of from 2 to 5 psi (0.01 to 0.03 MPa) is possible.

Our experience indicates use of a servo-controlled positive-displacement syringe pump provides the best back-pressure regulation capability. In this case two syringe pumps are used; one for flow-rate control and the other for back-pressure control. A constant prescribed back pressure can be obtained in two ways with this configuration. In one mode, the pore fluid pressure is established either using one of the pumps or an independent pressure generator. The flow-rate control pump is then activated for the prescribed flow rate and the back-pressure pump is activated (it pumps in an opposite direction to that of the flow control pump) and its rate adjusted manually until the fluid pressure achieves the desired steady state value. Usually, a perfect steady state is not achieved but the time variation of the pressure is sufficiently small that the permeability measurements are not significantly affected (i.e. no transient pressure pulses). Alternatively, the back-pressure control syringe pump can be servo-controlled using the output of an absolute pressure transducer. The pump rate is adjusted through time by signals from the surveillance computer system in order to maintain a constant fluid pressure at the transducer. The limiting factor of this latter method is the pressure resolution of the transducer, which is on the order of several 0.01 MPa.

In our system, back-pressure is controlled by use of either a servo-controlled syringe pump or adjustable spring-loaded pressure relief valve. The relief valve can be "valved out" of the system if necessary, but usually it is not and thus serves double duty as a safety pressure relief.

Permeability Measurement Systems

Continuous flow methods are appropriate for permeabilities as low as 0.1 to 1.0 microdarcy, provided sufficiently low, volume-flow rates can be achieved. For lower permeabilities, transient pulse-decay techniques are more appropriate. Summaries of both the theoretical and practical aspects of the transient-pulse method are provided by Hsieh et al (1981), Neuzil et al (1981), and Trimmer (1982). The basic components and requirements for both methods are discussed briefly.

Continuous Flow Methods: The basic objective of this method is to establish a steady-state fluid flow through the sample and measure the differential hydraulic head across the sample for a known volume-flow rate. Permeability is calculated assuming Darcy's Law and appropriate fluid properties (viscosity and density). The ideal configuration is to maintain either a constant flow rate or hydraulic head difference across the sample and measure the corresponding differential head or flow rate, respectively. The choice is usually dictated by the manner in which fluid flow is induced. The preferred method is to have controlled flow rate and measure the differential hydraulic head, which, in most instances, is equivalent to the differential pressure.

Servo-controlled positive-displacement syringe pumps provide a known flow rate and either two absolute pressure transducers (one upstream and one downstream) or a differential pressure transducer are used to measure the differential pressure. Use of absolute pressure transducers is more limited, especially for low flow rate through high permeability rock because of the limited pressure resolution of the transducers. Transducer resolution depends upon the pressure range of the transducer and the system electronics, but a reasonable limit is between 0.001 to 0.01 MPa (ca. 0.1 to 1.0 psi). Differential pressure transducers with changeable pressure plates offer higher resolution capabilities. Presently available, high line-pressures (80 MPa) differential pressure transducers are capable of resolving pressure differences as small as 1 MPa (0.0001 psi). Our system uses one of these transducers, hence we are able to measure high permeabilities (darcy range) even for very small flow rates (ca. 1 ml/hour).

Transient-Pulse Method: Continuous flow methods are not appropriate for permeabilities less than about a microdarcy. Instead, permeability is best determined by measuring the time decay of a fluid pressure pulse imposed across the sample. The permeability and, in some cases, the specific storage capacity, can be determined by either matching data curves against a family of theoretical curves or a nonlinear least-square fit of data to the theoretical equation. In order to use this method, the system must have the capability of imposing a prescribed pressure pulse across the sample and measurement, as a function of time, of the pressure difference using either two absolute transducers or a differential pressure transducer. We use a differential pressure transducer. The permeability system also must be designed so that the upstream and downstream storage capacities, S_u and S_d , respectively, are compatible with anticipated sample specific storage and permeability, as well as provide for a reasonable time duration of individual measurements. For design purposes, estimate of time for 90% decay of a pressure pulse is given by:

$$t_{90\%} \sim \frac{\mu L S_u}{k A}$$

where μ is fluid viscosity, L and A are sample length and area, respectively, k is permeability and S_u is storage capacity of the upstream reservoir. A large S_d/S_u ratio is often advantageous because the downstream pressure remains essentially constant during the pressure-pulse decay.

Jacketing Materials

Choice of sample jacketing materials is determined largely by temperature, but corrosion considerations become important if metal materials are used, especially at the elevated temperatures. For temperatures up to between 200° to 250 °C, Teflon and Viton (products of DuPont) are available. Gobran et al (1981) report that Viton started to thermally degrade at temperatures less than 200°C. At temperatures in excess of 250°C, Kalrez (product of DuPont), a fluorocarbon compound, is the only elastomer currently available. Kalrez is reported to be chemically stable up to temperatures between 260° and 315°C, depending upon the specific chemical formulation. Unfortunately, at this time, this material is very expensive. Consequently, at temperatures in excess of 250°C, metal jackets are the best candidates. In the absence of corrosive fluids, copper and lead are the two popular materials, but lead melts at 300°C. Copper is not suitable for use for most corrosive fluids, but gold plating of the interior of the jacket may reduce the corrosion problem. If copper can be used, then jacketing costs can be greatly reduced because extruded seamless copper tubing is readily available. Gold jackets are an excellent but expensive alternative. It has the advantage over Kalrez in the temperature range 250° to 300°C, because the gold jackets can be reprocessed. Extruded seamless gold tubing is available for outside diameters up to 2.5 cm. (1 inch); larger diameter jackets usually require "seamless" welding of gold foil.

If standard sized extruded metal jackets are utilized, sample diameter and end pieces should be sized with respect to the inside diameter of the tubing. Because of the desirability of using temperatures in excess of 250°C, we size our samples with respect to 0.75 inch gold tubing. This requires samples have a diameter of 0.725 inch. Diameter core barrels can be purchased for this size core.

Servo-Control and Data Acquisition System

Use of computer servo-control and data acquisition provides the system a high degree of flexibility and control of important experimental parameters. The flow rate and back-pressure control syringe pumps that we utilize have sufficient built-in rate controls that computer servo-control of the pumps is not needed under normal operating conditions. This capability is necessary, however, if we are to maintain a constant differential pressure across the sample; in this case, flow rates of the pumps would have to be adjusted to meet changes in permeability. Details of the computer system are presented in a subsequent section.

Heating and Temperature Systems

The pressure vessel is heated externally by strip heaters. Two temperature controllers are used to provide a zoned heating in order to minimize temperature difference along the vessel. An internal thermal couple is used to monitor sample temperature and control one set of the heaters. Experience of other workers indicates that use of an external heating system is favorable for establishing a

uniform temperature in the sample. Temperature differences will be less than 1°C. Tubing into and out of the pressure vessel are wrapped with heat tape and controlled by two separate controllers. Thermal couples are located at a number of points along the flow path to monitor and assure desired temperature homogeneity.

Fluid Flow-Rate Measurement System

Although the syringe pumps have built-in flow rate control and are calibrated, it is desirable to measure the fluid flow rate directly. The simplest and most effective way, especially at very low flow rates, is to measure the pressure difference across a length of small inside-diameter, high-pressure tubing. The tubing constitutes a "constant" permeability element and the equations associated with Poiseuille flow through a pipe can be used to relate the differential pressure measured to a flow rate, once the tube is calibrated. The pressure difference is measured using a high resolution differential pressure transducer of the type discussed earlier. Change of tubing length and/or diameter allows measurement of a wide range of flow rates. If a very small aperture tubing is utilized, it is essential that an in-line filter be positioned upstream of the flow gage. Coiling of the tubing affects the pressure/flow-rate relationship as observed by Gobran et al (1981). This effect can be accounted for in the calibration if precise absolute values are critical.

DESCRIPTION OF PERMEABILITY SYSTEM

Overview

After extensive study and analysis, an economical system design was finalized and the system constructed. Subsequently, several modifications were made during the proof testing stage. The system has been designed to allow a high degree of flexibility as well as permit measurement of permeability from the darcy range to the nanodarcy range at a wide range of physical conditions using geologically meaningful, chemically reactive, pore fluids.

A schematic diagram of the system is shown in Figure 1. A brief overview follows with a more detailed description provided subsequently. Fluid flow is generated by an upstream servo-controlled positive-displacement syringe pump that can be operated to generate either a constant prescribed flow rate or a constant differential pore-pressure across the sample. Under constant flow-rate control, the system can achieve flow rates as low as 0.2 ml/day and as high as 4800 ml/day at fluid pressures to 20 MPa. Back-pressure can be regulated by either a second, downstream servo-controlled syringe pump or an adjustable spring-loaded pressure relief valve. All wetted components (tubing, valves, tees, reservoir, specimen end pieces) subjected to elevated temperatures or in contact with corrosive pore fluids are made of the Ni-Cr-Mo alloy, Hastelloy C-276 (11,000 psi limit). All other tubing and components are 316 SS (11,000 psi pressure rating). A 55 ml, 30 cm long, tubular reactor made of Hastelloy C-276 serves as an upstream reservoir for corrosive fluids at elevated temperatures. A 2 micron, Hastelloy C-276, in-line filter is positioned immediately upstream of the sample, outside the pressure vessel. The sample, jacketed in either teflon or gold, depending upon the temperature, is

Figure 1. Schematic diagram of permeability system. See Table 1 for legend.

Table 1

Legend of symbols and abbreviations for Figure 1

Symbols:

2-way valve
3-way valve
absolute or differential pressure transducer
visual pressure gage
fluid reservoir
manual screw-piston pressure generator
filter
pressure tubing
electrical leads or cables
primary flow circuit
short circuit path
all components encompassed are in thermal chamber and all wetted components are constructed of HC-276

Abbreviations:

APT	: absolute pressure transducer
ARV	: spring-loaded adjustable pressure relief valve
DPT#	: differential pressure transducer
DPTI	: differential pressure transducer indicator
G1	: 3000 psi Heize gage
G2	: 60 psi gage (for calibration of DPT1)
G3	: 20,000 psi Heize gage
G4	: 3000 psi Heize gage
PS	: power supply for APT
PV1	: sample pressure vessel
PV2	: in-line tubular pressure vessel
PV3	: pressure vessel - supplemental downstream reservoir
R#	: fluid reservoir
RV	: pressure relief valve - rupture disc type (3000 psi limit)
T#	: tee
V#	: 2-way valve

hydrostatically loaded (up to 100 MPa) in an externally heated, high-pressure, Aminco hydrothermal reaction vessel modified to allow through flow of the pore fluid. The pressure drop across the sample is measured using a high resolution, variable range, differential pressure transducer capable of measuring a ΔP as small as 1 Pa to as large as 20 MPa (0.0001 to 2900 psi). Flow rate is measured using a flow gage constructed from high pressure capillary tubing and another high resolution differential pressure transducer. The system has a microcomputer for data acquisition and servo-control of the pumping rates of both the up- and downstream pumps. Permeability can be measured in either of two modes: (1) continuous, constant flow for high to medium permeabilities, or (2) pressure-pulse decay for permeabilities in the microdarcy to nanodarcy range.

Fluid Flow and Back-Pressure Systems

Controlled fluid flow and back pressure are achieved using two servo-controlled positive-displacement pumps manufactured by ISCO, Inc. (Lincoln, Nebraska). The model 314 pumps can generate constant-rate flow over a continuous range from 0.008 to 200 ml/hr at pressures to 20 MPa. The pumps have a 375 ml, Nitronics 50 (an Armco stainless steel with high strength and enhanced corrosion resistance) cylinder and a Hastelloy C-276 (HC-276) piston with pressure-actuated graphite-filled teflon seals. The pumps have a built-in servo-control unit to assure the constant flow rates. The built-in servo-control unit, however, can be overridden if another external servo-controller, such as a microcomputer, is utilized. The circuitry of the pump used for maintaining a back pressure was modified to allow controlled pumping in a reverse direction.

The basic flow path for fluid during normal operation is shown by the series of solid arrows in Figure 1. A rupture disc, fail-safe pressure relief valve (HIP, Inc. 3000 psi limit) is located immediately downstream of the upstream pump (P1). By opening V3, V12 and V13 and closing V8, the fluid pressure can be measured upstream of the sample by both the absolute pressure transducer (APT) (Sensotec, Inc., 35 MPa range) and the 21 MPa (3000 psi) visual Heize gage (G1). Conversely, if V8, V12, and V13 are open and V3 closed, then the fluid pressure is measured downstream of the sample. All of the components along the primary flow path between V4 and V7 are made of HC-276 and can be subjected to elevated temperature and contact with corrosive fluids. Furthermore, all components encompassed by the dashed-line box are contained within a large thermal chamber and are made of HC-276. Reservoir R2 is for the corrosive working fluid. A 55 ml, 30 cm long, vertically oriented, tubular reactor (PV2) (Pressure Products Industries, 35 MPa rating at 300 °C) is used as an in-line reservoir for the corrosive working fluid. During an experiment, distilled water delivered from P1 displaces the corrosive working fluid, which is typically more dense and occupies a lower position in the reservoir. From this in-line reservoir, the fluid passes through a 2 micron HC-276 in-line filter (F1) (Nupro "F" series, 21 MPa rating) in order to trap suspended colloidal particles that could plug the sample. At tee T2, tubing changes from 1/8" OD to 1/4" OD (1/8" ID) and the fluid pressure is communicated to the upstream side of the sample differential pressure transducer (DPT1), provided V17 and V19 are open. The fluid then passes into the pressure vessel (PV1) with the internal tubing coiling around and connecting to the lower sample end piece. The 1 m length of the coiled tubing assures the fluid will be of the same temperature as that of the sample. Details of the pressure vessel and the sample assembly are presented separately. The fluid flows upward through the sample and out of the vessel to tee T2. At T2, tubing changes back to 1/8" OD and

the fluid pressure is communicated to the downstream side of DPT1. From this point, the fluid passes through another in-line filter in order to trap suspended particles prior to entering the high-pressure, capillary tubing of the flow gage. A second differential pressure transducer (DPT2) measures the pressure difference across the flow tube, which can be converted to a flow rate. From the flow gage, the fluid either enters the reverse-stroking downstream syringe pump (if actuated) or exits the system into the effluent reservoir (R3) after passing through the adjustable, pressure relief valve (Nupro, R3A series, cracking pressures from 0.34 to 41.3 MPa). The relief valve can be isolated from the system (close V10), but usually it is left in with the cracking pressure set above that of the back-pressure maintained by the syringe pump and thus serves as a safety relief valve.

The dashed arrows (Figure 1) indicate a short-circuit flow path (open V3 and V8, and close V4 and V7), which allows the fluid to by-pass the sample. Amongst several purposes, this short-circuit facilitates flushing of the system and checking of the flow gage during an experiment without the interfering effects of the sample. The short-circuit configuration plus opening of V4, V6, and V7 permits one to subject the sample to equal pressures at both ends, which speeds up the process of pressure equilibration in a low permeability sample prior to a transient-pulse test. This configuration also is needed for calibration of transducer DPT1.

Pressure Vessel and Sample Assembly

The pressure vessel is a modified hydrothermal reaction vessel manufactured by Aminco. It is made of chrome-vanadium steel, has a 4 3/8" OD by 2 9/16" ID and is 10" deep. The maximum working pressure at 300°C is 110 MPa. To provide for fluid flow into and out of the vessel, a new pressure head was constructed, the basic configuration of which is shown in Figure 2. The 1/4" OD, HC-276 tubing passes through the pressure head with seals made internally using a single ferrule compression sleeve assembly (Autoclave Speedbite). Two high pressure, cone-and-thread connections are exterior for the confining pressure inlet and an internal thermal couple or other electrical lead through.

The sample assembly consists of: (1) an 0.725 inch diameter, variable length sample, (2) one or more sintered HC-276 porous discs at each end of the sample serving both as fluid spreaders and spacers, and (3) HC-276 end pieces (see Figure 3 for basic design). The pressure seal between the jacketing (either heat shrink Teflon or thin-walled metal tubing) and the 1° tapered, end pieces is achieved by arbor pressing, hemispherical metal rings over the jacketing and tapered end pieces. The HC-276 tubing is connected to the end pieces using standard cone-and-thread, high-pressure connections.

Confining Pressure System

The confining pressure system is of a simple manual type. Confining fluid pressure is generated by use of a manually operated screw piston pump (MPG2) (HIP, Co.) with a pressure rating of 210 MPa (30,000 psi). Confining pressure is measured using one of two visual Heize gages; a 21 MPa gage (G4) and 150 MPa gage (G3).

Top View
Pressure Head

Bottom View

Figure 2. Line drawings of the modified pressure head.

Figure 3. Line drawings of the sample end pieces.

Servo-Control and Data Acquisition System

Hardware: The system hardware consists of an AIM computer, an interface module, a cassette tape recorder, and a 3 channel, strip-chart recorder. The computer module houses the Rockwell AIM computer and Forethought Products Memory-Mate memory expansion, with the 8K BASIC interpreter ROM, 12K operating system ROM, 4K acquisition and control ROM, 40K RAM, and two 8-bit parallel ports; the Forethought Products STD-Mate interface and 2-slot STD bus; and the Data Translation 8-channel, 12-bit analog-to-digital converter card, with programmable full-scale input ranges of 10V, 1V, 100 mv, and 20 mv. The interface module contains the 8 analog input channel connectors, the RS-232 serial interface and connector, the programmable real-time calendar-clock, and the 13-bit digital-to-analog output converter.

Two low-level analog outputs from the interface module are connected to the LSCO pump control units. In the pump control units (when the MAN/REM switch is in the REM position) the control output signal is integrated over time to generate the actual pump rate; under steady-state conditions the control output signal will be zero and the pump speed will be some non-zero value.

Software: The set of special-purpose driver routines handle the input-output operations and transfer data to and from the AIM memory. The routines are designed to - drive the analog-to-digital converter and read the data from the 8 analog inputs

- read the date and time from the calendar/clock
- store data in memory and/or on tape
- read data from previously-recorded tape files
- generate an output voltage for pump control
- generate a linear time ramp and one-second time-out flags
- reset control, timing, and analog input parameters.

The servo-control output signals are generated by an interrupt program which, when activated, operates automatically and independently (i.e. a background mode) from BASIC programs. The magnitude of the control #1 output level is given by the sum of an error signal proportional to the difference between a reference analog input channel and a (user input) control set-point, and a derivative signal proportional to the difference between the current and previous error signals. The control #2 output level is determined only by an error signal (no derivative term). The proportionality constants for the error and derivative signals can be adjusted by the user. Control output #1 is normally used for the control of pore-fluid pressure, and control output #2 is used for the control of pore-fluid flow rate.

Permeability Measurement System

The key elements for measurement of permeability, in addition to the constant flow-rate syringe pump, are the differential pressure transducer (DPT1) (Validyne Eng. Co.) and a manually operated, piston screw pump (MPG1).

During a continuous flow experiment, V17, V18, V19 and V20 are open and DPT1 measures the differential pressure, ΔP , across the sample. Flow rate and differential pressure are recorded simultaneously either in analog form on the strip chart or in digital form by the computer and subsequent storage on the magnetic tape. These data provide the basic information, in addition to fluid properties, necessary for calculation of the permeability.

For a transient-pulse measurement, the sample may or may not be isolated from the remainder of the flow system depending upon the desired up- and downstream compressive storage capacities (proportional to up- and downstream volumes). The minimum upstream reservoir volume is 11 ml provided V6, V14 and V16 are closed; whereas the minimum downstream volume is 6 ml provided V7 and V21 are closed. By opening V6 and closing V5, the upstream reservoir can be increased to 66 ml which can further be increased to as much as 440 ml if V4 and V2 also are opened and the upstream pump cylinder is full. Similarly, the downstream reservoir volume can be increased from 6 ml to 190 ml by opening V21, which connects a 183 ml pressure vessel (PV3) to the fluid system. The downstream volume can be increased to as much as 570 ml if V7 and V9 also are open and the downstream pump cylinder is full. In general, it is desirable to have a much larger downstream reservoir than upstream reservoir so that the downstream pressure remains essentially constant during a pressure-pulse decay. The manual screw-piston, pressure generator is used to produce the upstream pressure pulse. Transducer, DPT1, measures the change of the pressure pulse with time with data being recorded in analog and digital form.

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