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MAGMA SIMULATION FACILITY DESIGN
CONSIDERATIONS, CAPABILITIES, AND
OPERATIONAL CONSIDERATIONS

MASTER

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MAGMA SIMULATION FACILITY DESIGN CONSIDERATIONS, CAPABILITIES, AND OPERATIONAL CONSIDERATIONS*

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ABSTRACT

The high-pressure, high temperature (60 Kpsi or 413.6 MPa/1600°C) facility described in this report is being used for molten rock (magma) experiments and metallurgical pressure bonding experiments. The unique design of the facility will accommodate samples as large as 10 cm dia. by 10 cm in height in an isothermal ($\pm 4^\circ\text{C}$ @ 1400°C and 44 Kpsi or 303.3 MPa) cylindrical hot zone. The facility history, capabilities, and operational considerations are thoroughly discussed with appropriate illustrations.

Since the system-contained energy while operating is approximately 1×10^6 ft-lbs (1.356×10^6 N-m) or 0.7 lbs (318 g) TNT equivalent, considerable discussion is related to hazards analysis and protection of the equipment and the operating personnel from damage caused by nonstandard conditions (interrupted H_2O flow, power outage, overpressure, etc.).

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MAGMA SIMULATION FACILITY DESIGN CONSIDERATIONS, CAPABILITIES, AND OPERATIONAL CONSIDERATIONS

Background

The need for a high pressure (60 Kpsi* or 413.6 MPa), high temperature (1600°C) facility for experimenting with large samples of molten rock was recognized in early 1975. The Sandia Magma Energy Program was in a fledgling state and an initial report ¹ had been published with recommendations regarding program direction. Negotiations with several manufacturers of internally heated pressure vessels and special pumping systems were begun in April, 1975. Complete system delivery, installation, and acceptance was delayed by materials availability and design problems until April, 1979.

The facility was designed to have unique pressure, temperature, and sample volume features for making basic properties measurements (viscosity, electrical conductivity, thermal conductivity) and materials compatibility determinations with various molten rock samples while simulating in situ magma conditions. A large (10 cm dia. x 10 cm ht.) uniform hot zone was required to accomplish these objectives. Secondarily, the facility could be used for hot isostatic pressing and diffusion bonding in various metallurgical experiments.

Approximately 25 experiments have been performed in the facility since April, 1979. These range from simple passive materials compatibility tests in sealed platinum capsules to attempts to make active viscosity measurements with geologic materials. Isostatic diffusion bonding tests have been performed with multiple samples being simultaneously compressed under controlled conditions.

*Primary units are those on equipment and in associated system documents.

The facility is located at the Building 9939 Test Site. The vessel, intensifier, and heat exchanger are in Building 9938-W and the controls are located in 9939, Room 1. The contained energy in the system when pressurized to 60 Kpsi (413.6 MPa) is 1×10^6 ft-lbs. (1.356×10^6 J) or the equivalent of 0.7 lbs. (318 g) of TNT. The facility was deliberately located at a remote site to minimize hazards to surrounding equipment and personnel. Figure 1, (a) and (b) illustrate these locations.

Design Considerations

Overall System - The original Request for Quotation (RFQ) solicited proposals from several manufacturers for the following system:

Sample Volume:	0.1 - 1.0 liter
<u>Corrosive Gas Working Pressure:</u>	60 Kpsi (413.6 MPa)
Optical Ports:	1 in lid and 2 on sides at 180°
Electrical Feedthroughs:	6 for furnace power and 12 for instrumentation
Pressure Ports:	4
Furnace Capability:	R.T. to 1600°C
Mechanical Sample Agitator:	Internal

After many months of evaluation of proposals and realistic compromise, Sandia contracted with Autoclave Engineers, Inc., for the vessel. Later Autoclave also was awarded a contract for the furnace. Harwood Engineering supplied the 2-stage intensifier. The system now meets the following criteria:

Fig. 1(a) Location Map

Fig. 1(b) Site Map

Sample Volume:	0.8 liter
<u>Inert Gas</u> Working Pressure:	60 Kpsi (413.6 MPa)
Electrical Feedthroughs:	2 for furnace power and 12 for instrumentation
Pressure Ports:	3 for high pressure connection
Furnace Capability:	R.T. to 16 0°C

No optical ports or mechanical stirring capabilities are incorporated into the vessel design due to strength and cost considerations.

Subsystems

Pressure Vessel - The vessel weighs approximately 5000 lbs. (2273 kg) and is 43" (109.2 cm) in length and 22" (55.9 cm) in diameter. It is mounted on a single point trunnion for ease of maintenance. The vessel has a 310 ss liner with 4322 high strength steel ring forgings around the HY140 inner strength member. The inner strength member has a shrink fit on the liner. The helical passage between the liner and the member provides a coolant path. The bottom end plug is A286 ss machined in one piece and the top end plug is 4340 with a 310 end cap. The large closure nuts are also 4340 steel.

A screw-thread lift mechanism, permanently bolted to the vessel, is used to raise and lower the bottom plug and nut assembly. This fixture is aligned on the vessel centerline for repeatable, correct closure.

Ultimate failure for the vessel has been calculated ² to be 120 Kpsi (827.3 MPa). The vessel was tested to 75 Kpsi (517 MPa) and is rated for continuous operation at 60 Kpsi (413.6 MPa) with a maximum tolerable temperature at the liner I.D. of 300°C. Figure 2 shows the vessel construction in cross section.

Fig. 2 Pressure Vessel Construction

Furnace - The furnace is primarily constructed from molybdenum. The heating elements are formed from sheet stock with milled slots. The sheet is then formed into 2 concentric cylinders. The resulting serpentine assembly is supported on boron nitride insulators and spacers. A 2-level pedestal allows the elements to be supported beneath the uniform hot zone. The deliberately convective design provides a very uniform ($\pm 4^{\circ}\text{C}$ at 1400°C and 44 Kpsi or 303.3 MPa) hot zone. This zone is capped with an insulating shield made from concentric metallic cylinders separated by layers of high temperature insulation.

The furnace elements are resistance heated via an SCR-controlled power supply operating on 480V AC and stepped down to 90 volts and 100 amps at the furnace. Temperature control is maintained by a Barber-Colman set-point controller. This controller has a remote mode that permits programming of the heating cycle from an external programmer. Figure 3 illustrates the furnace configuration.*

Intensifier - The Harwood 2-stage intensifier has a horizontal piston that is alternately driven from the low stage end to the high stage end by hydraulic fluid. The fluid system is isolated from the gas system by a series of sliding seals and isolation chambers open to the atmosphere. The Maximum Allowable Working Pressure (MAWP) for the intensifier is 60 Kpsi (413.6 MPa) using inert gas. The intensifier is not compatible with gases containing hydrogen or sulfur.

*The exact details of construction are the proprietary property of Autoclave Engineers, Inc.

Fig. 3 Furnace Construction

The factor of safety for the pumping system is 3.94:1 per Harwood Engineering ³ . The pump is a standard DA-4-6-60 that is special only in that the controls and readouts are located remotely. The hydraulic oil reservoir and pump chambers are provided with cooling jackets for water flow. Figure 4 is a block diagram of the intensifier.

Heat Exchanger - This is a McMaster-Carr liquid-to-air unit designed for 50 KBtu/Hr (14.7 kW) dissipation at 80°F (27°C) ambient temperature. The furnace system needs to dissipate approximately 34 KBtu/hr⁴ (10 kW) to remain within acceptable inner wall temperatures for the vessel. The coolant flow rate through the vessel is 2.5 gal/min. (0.1577 l/s) at 90 psi (0.6 MPa). The heat exchanger is located outside the 9938-W test cell and is connected to the vessel by flexible hoses. The exchanger reservoir contains 20 gals (76 l) of a 50/50 antifreeze/water mixture. The coolant loop is shown in Figure 5.

Acoustic Emission - A contact microphone is attached to the top of the pressure vessel. Signals detected by the microphone are amplified by a preamplifier and sent to a counter. The counter then sends the signal to a digital to analog converter that processes the signal and permits an analog display of events. The normal acoustic signals detected may be produced by the movement of o-ring seals, expansion and contraction of the vessel walls, and noise created by thermal expansion of the furnace elements. A departure from this normal background noise could signal a mechanical failure of the vessel or a leak developing at a seal. A block diagram of the acoustic emission system is shown in Figure 6.

Fig. 4 Intensifier Block Diagram

Fig. 5 Coolant Loop

Fig. 6 - Acoustic Emission System

Interconnections - There are three large patch panel cabinets (A, B, & C) located in Building 9939, Room 1 and three interconnecting and corresponding cabinets at Bldg. 9938-W. The cabinets are connected by underground conduit. Cabinet A contains terminal strips for circuits related to building functions (alarms, gas supply pressures, and television monitoring). Cabinet B contains connections for operating apparatus during experiments (pumps, furnace, vents, water flow valves, etc.). Cabinet C contains only connections related to low level signals from the experiment (thermocouples, pressure transducers, and acoustic emission). This cabinet has panels for 10 type K and 8 type B thermocouple connectors plus 30 RF connectors.

The hardwired cables between the computer signal interface in 9938-E and the computer console in 9939/Room 1 also pass through Cabinet C. Figure 7 is a block diagram of the interconnection panels.

Interlocks - Proper coolant flow through the pressure vessel is sensed by two series - connected flow switches as the coolant exits the vessel jacket. The appropriate contacts of the switches are used to operate a multiple contact relay and to activate personnel warning beacons (one inside and one outside the 9938-W test cell). The relay contacts are used to interrupt furnace control power and to open or close appropriate solenoid-operated city water valves for emergency cooling purposes. In the event of a power outage the same flow switch fail-safe sequence takes place to power down the experiment and convert to an emergency cooling mode. These circuits are equipped with latching features that prevent a return to a powered state when flow or power is restored.

Fig. 7 - Interconnections Diagram

Another flow switch is located downstream from the previously mentioned switches to provide a latching option for the heat exchanger in the event it malfunctions for any reason and then tries to restart.

Additional protection for the furnace system is provided by yet another interlock that uses a dial-type pressure gage with adjustable electrical contacts that are triggered by the passage of the gage needle. This interlock prevents furnace power from being turned on until the vessel pressure is above the gage set point. This circuit also includes a latching feature that prevents the furnace from being re-engaged once the interlock has been triggered.

The furnace control panel has a latching circuit that will not allow power to be restored in the event of power outage until the circuit is manually reset. This protects the furnace element from receiving a large power surge. Figure 8 shows the interrelation of the interlocks.

Capabilities

Present

Pressure - The system is rated at 60 Kpsi (413.6 MPa) but also has a selection of rupture discs that can be installed to positively limit pressure for various experiments. The intensifier controls provide pressure regulation within $\pm 1\%$ of the set point. An automatic vent valve is included in these controls. Usually, the vessel is pre-pressurized to a moderate pressure, the furnace is turned on, the gas is expanded, increasing the pressure, and then the pressure is topped off to the desired value.

Fig. 8 - Protective Interlock Diagram

A vacuum roughing pump is used to evacuate the vessel to 20 microns (10^{-6} meters) after each closure of the vessel. This is necessary to eliminate oxidation of the furnace elements during heating. For good temperature control, the initial pressure should be above 3 Kpsi (20.7 MPa). This permits good thermal communication between the control thermocouples and the furnace elements.

Pressure is monitored by several transducers on the intensifier, a transducer on the vessel, and 2 Bourdon-tube dial gages on the vessel. The dial gages are read via the television monitoring system.

The intensifier is strictly limited to inert gases and argon is normally used as the pressure medium. The pressure vessel is hydrogen compatible but extensive analysis of operating procedures should be performed before any hydrogen is used in an experiment. Figure 9 shows typical pumping rates.

Temperature - The design temperature is 1600°C. The system has been taken to 1400°C at 44 Kpsi (303.3 MPa). None of the experiments have yet required the maximum temperature. During the acceptance tests (1400°C/44 Kpsi or 303.3 MPa), the temperature distribution in the 10 cm dia. x 10 cm hot zone was $\pm 4^{\circ}\text{C}$. Type B (Pt-30Rh/Pt-6Rh) thermocouples are used, and the temperatures are displayed on Doric readouts as direct readings in degrees C. Control thermocouples for the furnace are dedicated to that function and the experiment thermocouples are placed as needed for the experiment.

A Data-Trak drum programmer is currently being used to bring the temperature to approximately 1100°C. Above 1100°C manual control is used. Figure 10 is a plot of typical heating rates.

Fig. 9 - Typical Pumping Rates

Fig. 10 - Typical Heating Rates

Sample Size - The uniform hot zone is 10 cm. in diameter by 10 cm. in height. An Al_2O_3 catcher crucible is used in every experiment to prevent sample spillage onto the furnace elements in the event that a sample crucible breaks. This catcher crucible has an inside diameter of 8.25 cm. and an inside height of 9.52 cm. One large sample may be placed in the sample crucible or many small samples can be run simultaneously (typically, small sealed Pt tubes containing various proportions of powdered rock, organic dopants, and water).

Quench Option - The cooling rate of a sample depends on sample thermal mass, surface area, temperature, and also gas density of the pressurizing medium. The intensifier does not have the pumping capacity to compensate for the pressure loss due to gas cooling. An auxilliary pressure vessel (2.779 l) is used to ballast the pressure loss. This vessel has a MAWP of 60 kpsi (413.6 MPa), can be pre-pressurized to an appropriate level and then allowed to equilibrate with the sample vessel. The quench vessel is located in 9938-E and the rate of equilibration is controlled manually. Figure 11 shows typical cooling curves.

Acoustic Emission - This accessory may be used to monitor vessel behavior or to listen for a particular discrete event that occurs inside the vessel (release of a metallic sphere for viscosity experiments or the onset of flow when venting the vessel). The acoustic signature of the event must be established as a prerequisite so that it can be discriminated from the background noise.

Fig. 11 - Typical Cooling Rates

Data Acquisition - Temperature and pressures are recorded on a Bascom-Turner 8120 programmable recorder. This instrument utilizes a floppy disc for programming and data storage. The data can be smoothed and manipulated post-test by retrieving it from the disc. The data sampling rate is variable for different types of experiments and 8 channels of data can be taken. One channel of data can be plotted on a real-time basis.

Data is also periodically recorded manually during the experiment in a system log book. Data of particular interest in this log are temperatures and pressures in auxilliary portions of the test system.

Remote Television Monitoring - A remotely operated television system with pan, tilt, zoom, and focus capabilities is available for reading auxilliary instruments associated with nonstandard experiments. This system is normally used to read dial gages in the test cell from the control room.

Remote Audio Monitoring - A master intercom station is available in both the control room and the test cell. Voice communication can be established from either end during pre-test checkout of the system. Also, typical operating sounds of the vacuum pump, intensifier, and power relays can be detected during an experiment and monitored for changes that might indicate potential problems.

Future

Computer System - A Hewlett-Packard 1000 system has been purchased for data acquisition and control. It is operated via a graphics terminal and has high speed printing and hard copy capabilities.

When the total system is eventually converted from manual control to computer control, the data acquisition will be transferred first and then the control function. See Figure 12 for details of the computer system.

Volatile Injection and Circulation - This capability has been a goal of the Magma Energy Research Program for several years. The difficulty in designing, constructing, and implementing this capability arises from the chemical incompatibility between the candidate gases (CO , CO_2 , SO_2 , H_2S), the existing intensifier, and the molybdenum furnace elements. A bladder arrangement must be used that will permit the gases to be in contact with the sample material and permit the argon gas to act as an external supporting medium for the bladder. This bladder would probably be made out of thin platinum sheet and typical test temperatures would be 1100° - 1400°C . It is readily apparent that extremely accurate differential pressure control is needed to prevent rupture or collapse of the bladder.

Preliminary analysis of this capability was made by Northrup and Baudoin ⁵ in the mid 1970's; Greenholt and Wemple ⁶ in the late 1970's; and most recently by Wemple and Wawersik ⁷. The latter believe that the task is "doable" based on design work by Wawersik ⁸ on another research program. The concept involves a frictionless accumulator, precision solenoid control valves, and differential pressure transducers. Figure 13 is an illustration of the most detailed system concept yet developed. The cost of implementing this

Fig. 12 - Computer System

Fig. 13 - Corrosive Gas Pressure Balancing Concept

capability is estimated to be \$50K - \$75K, a significant cost that must be included in future Magma Energy Research budgets if the concept is to become a reality.

Operational Philosophy

Safety - As mentioned previously, when pressurized to 60 Kpsi (413.6 MPa) the system contains 0.7 lb. (318 g) TNT energy equivalent. It is obvious that extreme caution must be used in operating the system to protect site personnel and minimize property damage in the event of system breaching. The system Pressure Safety Analysis Report (PSAR) details the design analysis, predicted failure modes, proof test, and use considerations. The whole facility is shown in block diagram in Figure 14.

A "buddy system" approach for operating personnel forces each operational decision to be approved by both operators before it is executed. The system Safe Operating Procedure (SOP) requires the use of protective equipment (safety shoes, glasses, etc.) by the operators and lists a typical operating sequence. Emergency shut-down procedures are also contained in the SOP.

The 9939 Test Site SOP requires various levels of personnel protection corresponding to various hazard levels. The requirements range from simple audible warnings to beacons and physical barriers. All experiment SOP's at the site must be reviewed by the site Safety Officer and cannot be in conflict with the overall site SOP.

Typical Experiments

Short-Term - These tests are arbitrarily defined as those that can be completed in a normal work day. However, several days of

- 1 - Hazard Warning Equipment
- 1A - Access Monitoring System
- 2 - Acoustic Emiss. Det./Amplifier
- 2A - Acoustic Emiss. Dis./Counter
- 3 - Computer Remote Interface
- 3A - Data Acq/Cont. System
- 4 - Vessel/Furnace Assembly
- 4A - Furnace Pwr. Supply
- 5 - Inert Gas Intensifier
- 5A - Intensifier Control

- 6 - Argon Supply
- 7 - Coolant System
- 7A - City Water System
- 7B - Recirculating System
- 8 - Vac. Roughing System
- 9 - Press./Temp. Display
- 10 - TV Camera
- 10A - TV Monitor
- 11 - Intercom
- 11A - Intercom

Fig. 14 - Total System Block Diagram

preparation may be required prior to the test. Metallurgical diffusion bonding and hot isostatic pressing; and geological sample melting, preform preparation, and viscosity measurements are typical short-term experiments.

Long-Term - These tests can be defined as requiring more than a normal work day for completion. Since the equipment would not be manned 24 hrs./day, the experiment must be carefully designed to minimize failures that would cause automatic shutdown of the system. Typical of these experiments would be materials compatibility, injection of volatiles into molten rock, and energy extraction studies with heat exchangers.

Interlock Testing

As mentioned previously in this report, the protective interlock system is quite extensive and complicated. Each interlock system must be periodically exercised to determine that it is working properly. This must be done by qualified operators working in the "buddy system" mode to minimize damage to the system.

System Maintenance

Maintenance should be performed in accordance with the manufacturer's recommendations for each subsystem. These recommendations are straight forward in terms of lubrication, seal replacement, checking torque values, etc. However, since the subsystems form the total system and are interconnected by wiring, hoses, pipes, solenoids, etc., the operators must be alert to problems that develop and/or symptoms that indicate a malfunction or potential malfunction. In summary, the system must be continuously

monitored by experienced operators to assure that a simple breakdown does not effect a program schedule or jeopardize personnel safety.

Summary

The Magma Simulation Facility has been operational for 18 months. During that time, approximately 25 experiments have been performed at various pressures and temperatures. The system has performed reliably and has not required any unusual or costly maintenance. Temperature and pressure performance is within the manufacturers' claims.

The system has been upgraded to include a combination of protective interlocks to minimize damage to equipment and/or hazards to operating personnel.

Several future capabilities are being explored and will be integrated into the system when the designs are firm and adequate funding is available.

Future successful operation must be closely tied to well-planned experiments, preventative maintenance, and complete awareness by the operating personnel of the system performance criteria.

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