

SANDIA REPORT

SAND99-1031

Unlimited Release

Printed May 1999

Description of a Furnace for the Creation of Anisotropic Porous Metals

David T. Schmale, Mark Miszkief, and Thomas Gutsch

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

Approved for public release; further dissemination unlimited.



Sandia National Laboratories

RECEIVED
JUN 09 1999
OSTI

Issued by Sandia National Laboratories, operated for the United States Department of Energy by Sandia Corporation.

NOTICE: This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government, nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors, or their employees, make any warranty, express or implied, or assume any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represent that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government, any agency thereof, or any of their contractors or subcontractors. The views and opinions expressed herein do not necessarily state or reflect those of the United States Government, any agency thereof, or any of their contractors.

Printed in the United States of America. This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from
Office of Scientific and Technical Information
P.O. Box 62
Oak Ridge, TN 37831

Prices available from (703) 605-6000
Web site: <http://www.ntis.gov/ordering.htm>

Available to the public from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Rd
Springfield, VA 22161

NTIS price codes
Printed copy: A03
Microfiche copy: A01



DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

SAND 99-1031
Unlimited Release
Printed May 1999

DESCRIPTION OF A FURNACE FOR THE CREATION OF ANISOTROPIC POROUS METALS

David T. Schmale and Mark Miszkiel
Materials and Process Sciences Center
Sandia National Laboratories
P.O. Box 5800
Albuquerque, NM 87185

Thomas Gutsch
Thermal Technology, Inc
1911 Airport Blvd.
Santa Rosa, Ca. 95408

Abstract

A furnace has been built for the purpose of producing anisotropic porous metals through solid-gas eutectic solidification. This process allows control of continuously formed anisotropic pores in metals and was discovered at the State Metallurgical Academic University in Dnepropetrovsk Ukraine. The process incorporates hydrogen gas within the metal as it solidifies from the molten state. Metals which do not form hydrides, including iron, nickel, aluminum, copper and others can be formed in this manner.

The furnace is housed within a $\sim 0.64 \text{ meter}^3$ (30 ft³) ASME code stamped cylindrical stainless steel vacuum/pressure vessel. The vessel is a water chilled vertical cylinder with removable covers at the top and bottom. It can be evacuated to 20 mTorr or pressurized to 5.5 MPa (800 psi). A charge of 2700 cc (167 in.³) of molten metal can be melted in a crucible in the upper portion within a water cooled 30 cm (12 in.) ID induction coil. A 175 kW Inductotherm power source energizes the coil. Vertical actuation of a ceramic stopper rod allows the molten metal to be tapped into a solidification mold beneath the melting crucible. The cylindrical mold rests on a water cooled copper base inducing directional solidification from the bottom. Mixtures of hydrogen and argon gases are introduced during the process. The system is remotely controlled and located in a structure with frangible walls specially designed for possible ambient pressure excursions as a result of equipment failure.

This paper includes a general description of the furnace and operating procedure and a detailed description of the control, monitoring and interlock systems.

this page intentionally left blank

Introduction

The anisotropic porous metals casting furnace was built to create engineering materials that have superior strength to weight ratios as compared to their solid non-porous counterparts. The process, called Gasar (an acronym taken from the Russian term for this process), is based on solid-gas eutectic solidification of a molten material saturated with hydrogen. This furnace, the first of its size in the United States, was designed and constructed for the purpose of producing Gasar materials on an industrial scale.

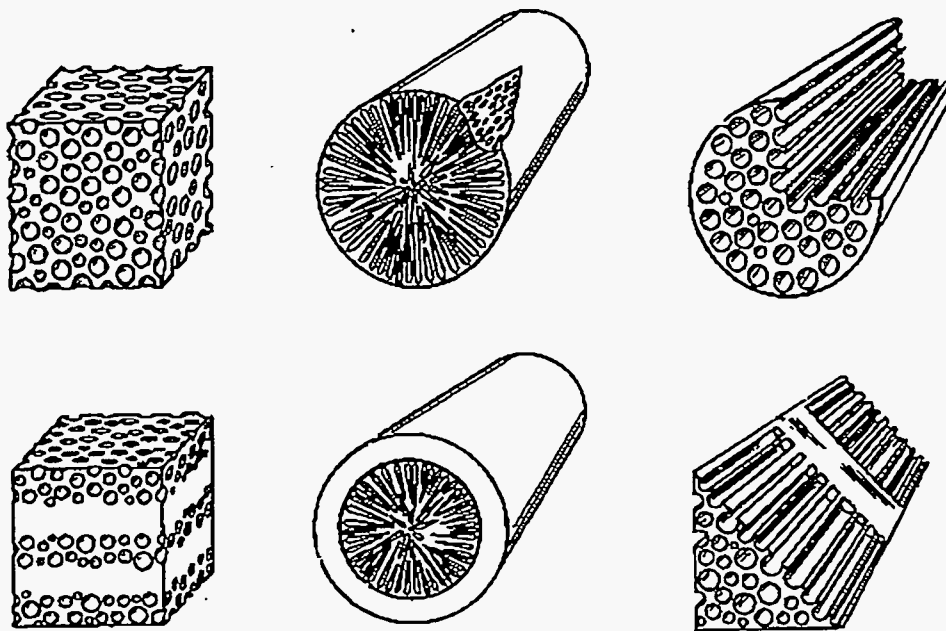


Figure 1. Possible Gasar structures. Source: Shapovalov, US patent No. 5,181,549, Jan, 1993.

To form the Gasar material, pressurized hydrogen is introduced into the molten metal. During casting, the rate of the solidification front is controlled by manipulation of melt superheat and pressures during melting and solidification so that the hydrogen emerges from the melt to form anisotropic pores in the solidified material. Anisotropic structures are those which have physical structures and properties that have different measurements along different axes. For cylindrical shapes with pores parallel to the axis of the cylinder, casting mold are designed to direct the solidification front from the bottom up, away from a chilled copper plate. For cylindrical, tubular shapes with radial pores perpendicular to the centerline, the solidification front is directed radially from the inside diameter of the tube away from the center. **Figure 1** illustrates the wide range of possible structures.

These materials have applications such as bearing, filter materials, lightweight structures, catalytic substrates, crush absorption and vibration damping. Gasars will compete with traditional methods of manufacturing porous materials, such as powder metallurgy, and they also offer a class of materials with unique properties.

Furnace Description

Operation of the furnace with hydrogen at pressures up to 5.5 MPa (800 psi) dictates that the system be operated remotely from a control room behind a steel reinforced concrete wall. The layout of the furnace, control room, gas house and power supply is shown in **Figure 2**. The exterior structure walls surrounding the furnace are frangible. The operator has control of the induction power supply, the chill water system, vessel pressure and the stopper rod actuator. Remote operation of the furnace requires the use of automatic interlocks for the gas, chill water, vacuum systems and induction power supply.

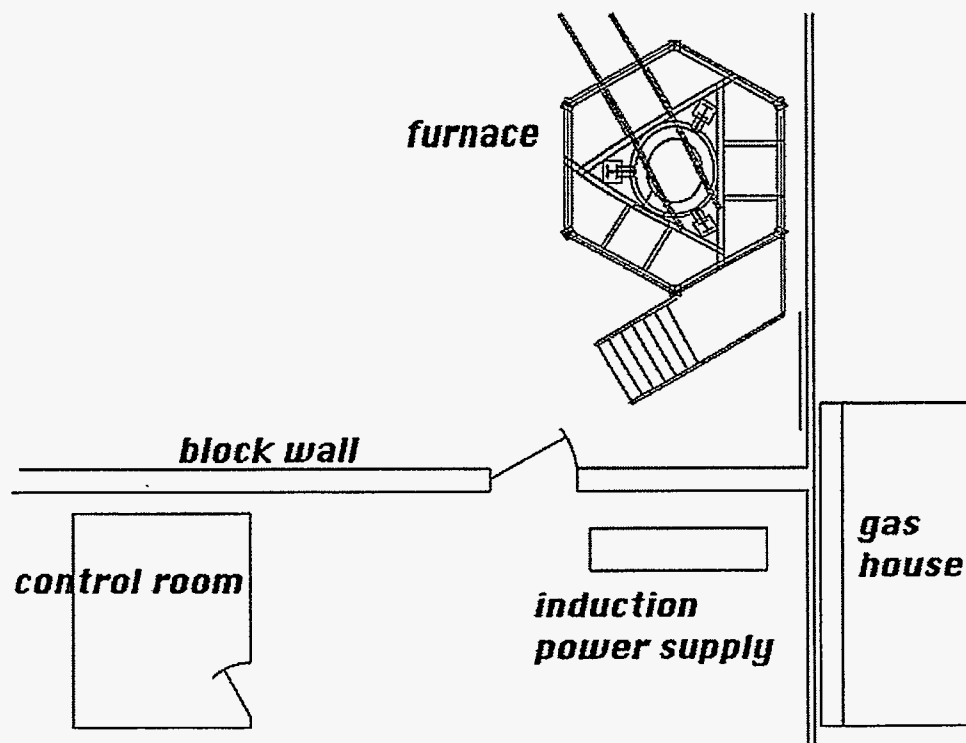


Figure 2. Plan view of system layout.

Approximately 36 individual conductors (not including the induction power supply, the 8 thermocouple circuits or the video lines) connect the control console to the furnace.

Video cameras provide real-time views of the upper melting section, through a viewport, and the lower casting section from inside the furnace. The internal camera was designed for high pressure underwater applications by Outland Technology, Inc., and was modified for this operation with a chill water coil and a quartz lens. Electrical feedthroughs for the internal thermocouples and video were manufactured by Pave, Inc.

A schematic section diagram of the furnace, **Figure 3**, shows how the components fit into the ASME certified pressure vessel. The final code complying design and manufacture of the vessel was performed by Victoria Machine and Fab, Inc., of Victoria, Texas. The upper lid is removed via a lifting system suspended from a hoist integral to the building structure.

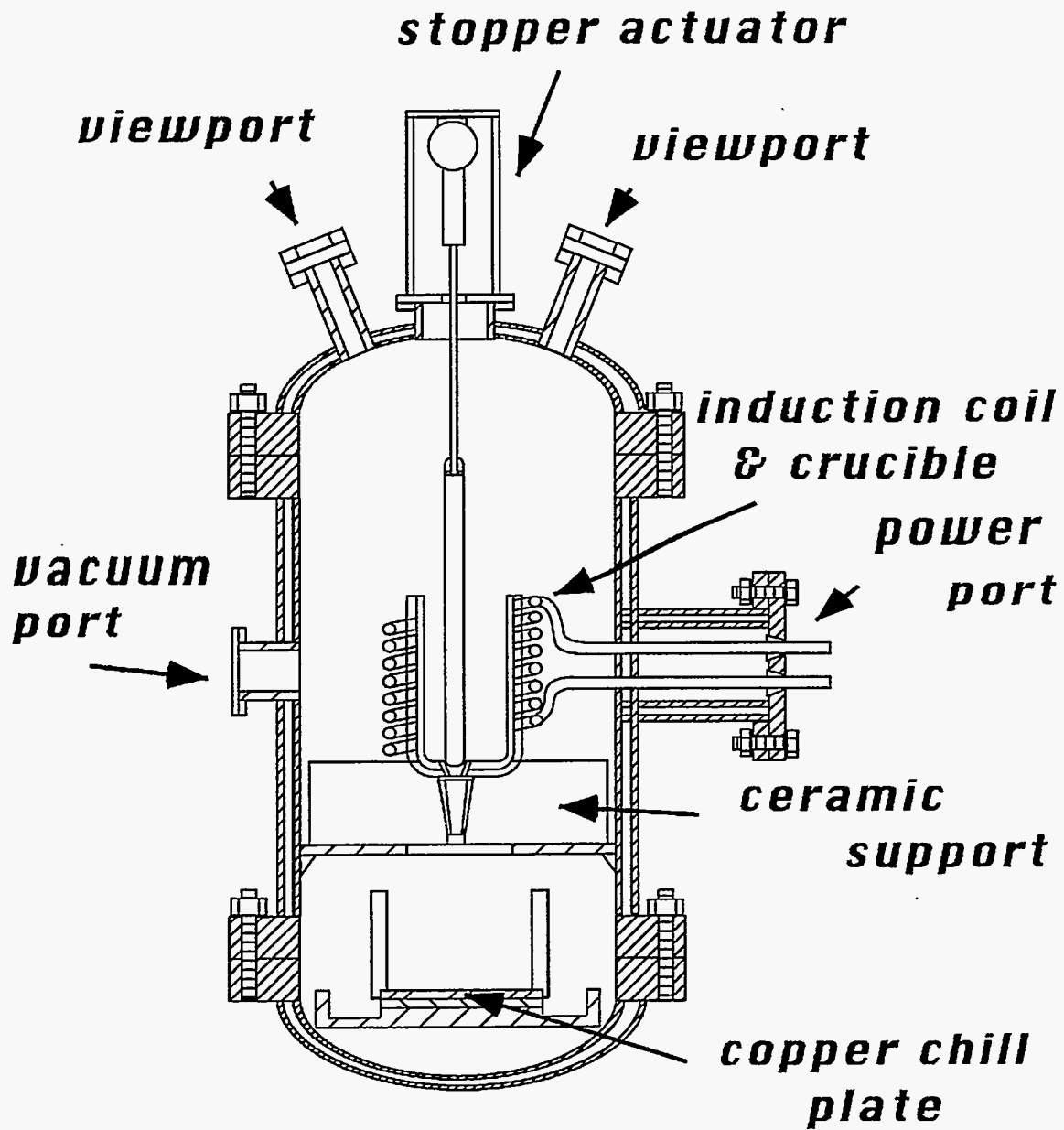


Figure 3. Section view of the Gasar casting furnace.

The lower lid is raised and lowered on a Rol-Lift, Inc. hydraulic scissors lift table modified with wheels and a track that allows the assembly to be rolled out for easy access. The furnace is shown in **Figure 4** surrounded by a platform constructed for easier access to the melt crucible and the monitoring devices.

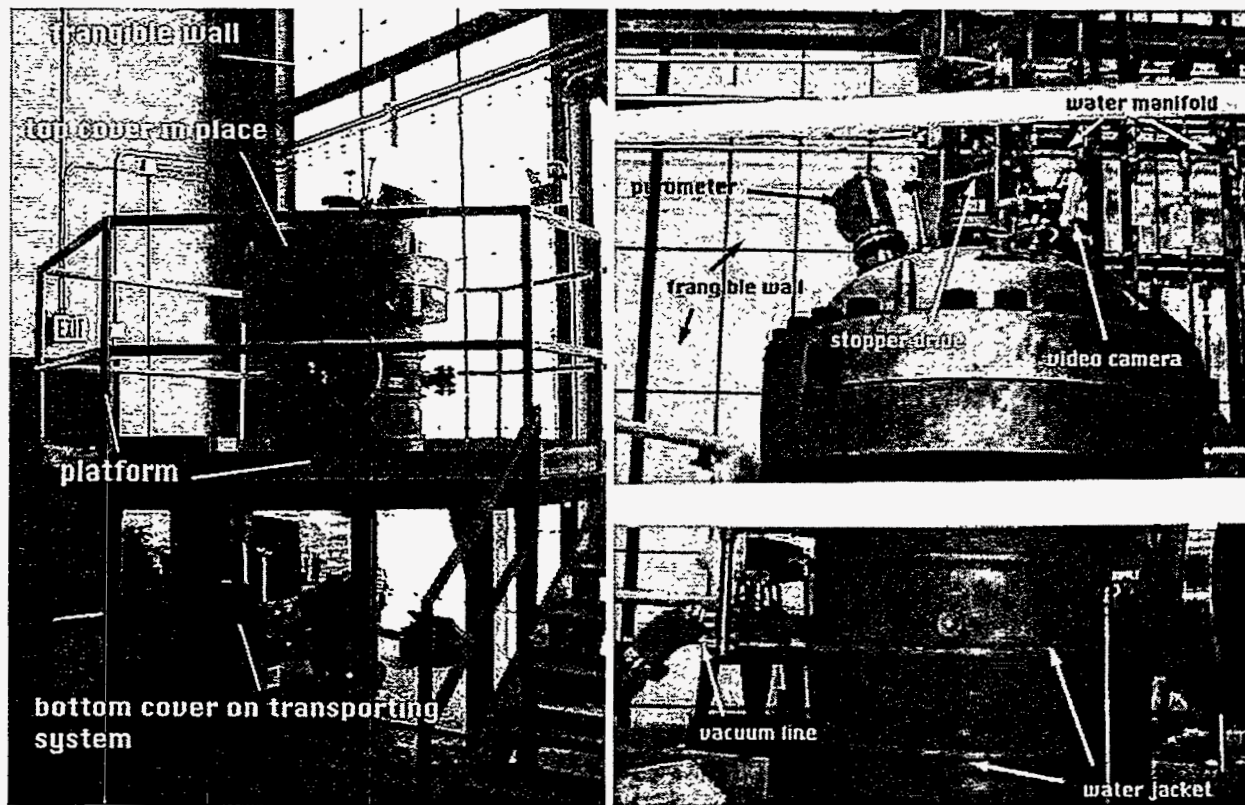


Figure 4. left: The furnace surrounded by the access platform with the lower cover lowered on the scissors jack and rolled out for access. Right: Upper furnace showing stopper rod actuator, pyrometer and the video camera that views the melt.

Metal is melted in a ceramic crucible by a four-legged Inductotherm coreless induction melting coil. The 25.4 cm (10.0 in.) diameter by 30.4 cm (12 in.) coil, shown in **Figure 5**, is wound from 19 mm (0.75 in.) OD heavy copper tubing.

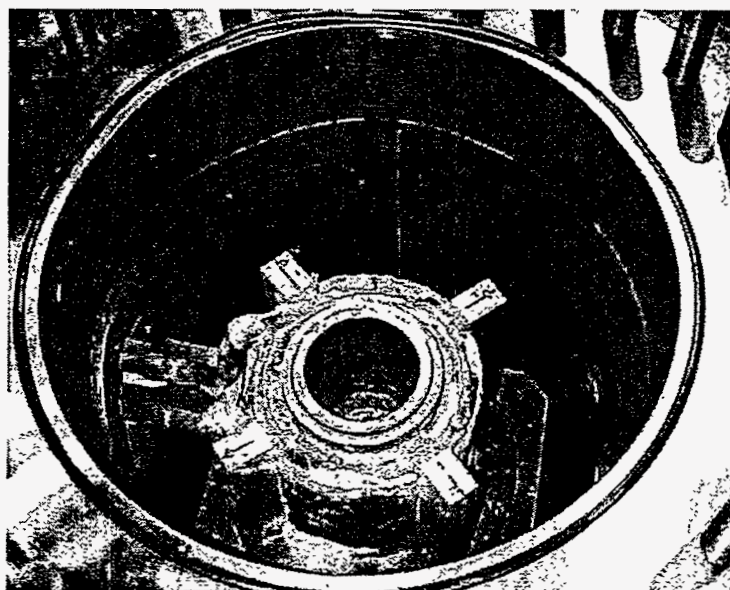


Figure 5. Induction coil inside furnace shown completely encapsulated in a hardened ceramic slurry. The Blasch double walled crucible is fitted into the encapsulated coil.

An Inductotherm 175 kW induction power supply (**Figure 6**) provides alternating current at 3000 Hz (650 amps max) and less than 550 volts to the coil. Ceramic feedthroughs in the power port, designed to withstand the pressures in the furnace, were also designed by Inductotherm, Inc. Controls for the power supply are located both in the control room and on the power supply. The Blasch alumina bottom pour double lined crucible is located at the center of the induction coil and supported by a ceramic pedestal. The crucible can hold 2700 cc (167 in.³) of molten metal and employs a custom made alumina stopper rod to seal the pour spout and prevent leakage of molten metal.

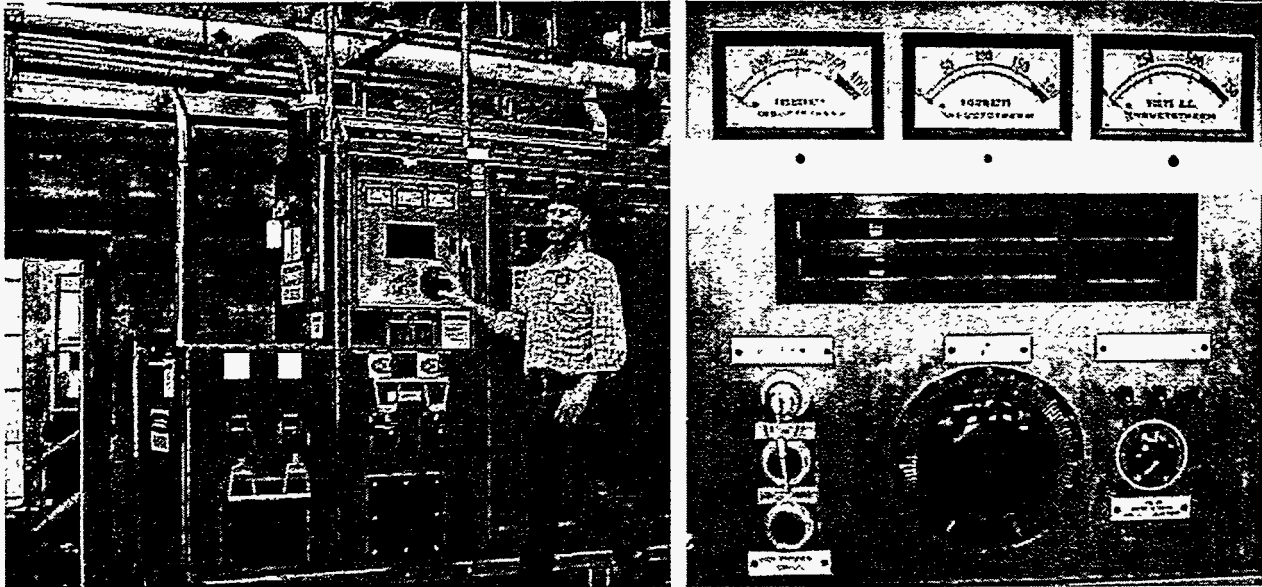


Figure 6. Photographs of the Inductotherm 175 kW power supply and the control panel located in the control room. (One of the system builders generously provides a size reference.)

A universally jointed stainless steel lift rod with an Inconel 718 radially sprung gripper attaches the stopper rod to the actuator. The 445 N (100 lb.) capacity stopper rod actuator is mounted on the upper cover of the furnace and manufactured by Duff-Norton, Inc. The actuator is shown in **Figure 7** and is limited to 50 mm (2.0 in.) travel by proximity switches mounted in the frame. A greased and polished section of the rod slides through a compressed o-ring to form a gas-tight seal with the furnace cover.

The vessel, with 22 mm (0.875 in.) thick stainless walls, is designed to operate at 5.5 MPa (800 psi) at an internal temperature of 230°C (450°F) and was proof tested to 8.25 MPa (1200 psi). Since the vessel is evacuated and backfilled before use to ensure the removal of oxygen prior to operation, the vessel was also vacuum leak checked by the manufacturer. The center and upper sections of the vessel (**Figure 4**) are double-walled to provide a chill water jacket rated to .86 MPa (125 psi) working pressure. The lower cover is below the heat zone and is protected from molten metal with a massive secondary containment housing/moat system that also locates the casting molds. The upper video camera and the pyrometer are mounted above two sightglass ports shown in **Figure 8**. The sightglasses, a 10 cm (4 in.) diameter and a 5 cm (2 in.) diameter are manufactured by Pressure Products Company, Inc.

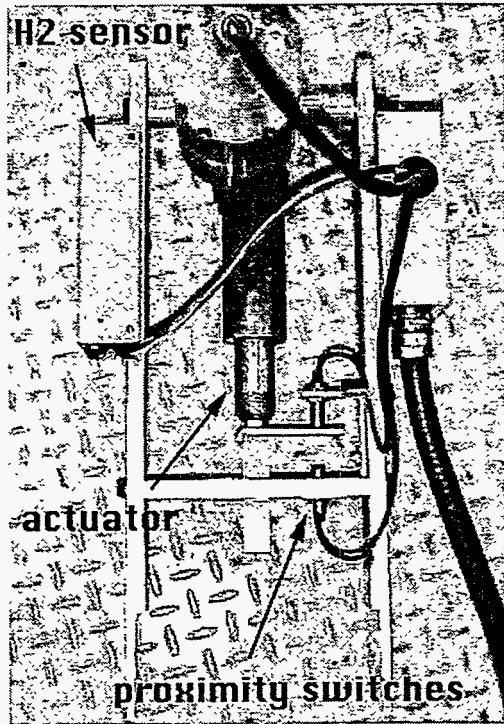


Figure 7. Dayton screw driven actuator in framework with proximity switches that limit travel to 50 mm (2.0 in.).

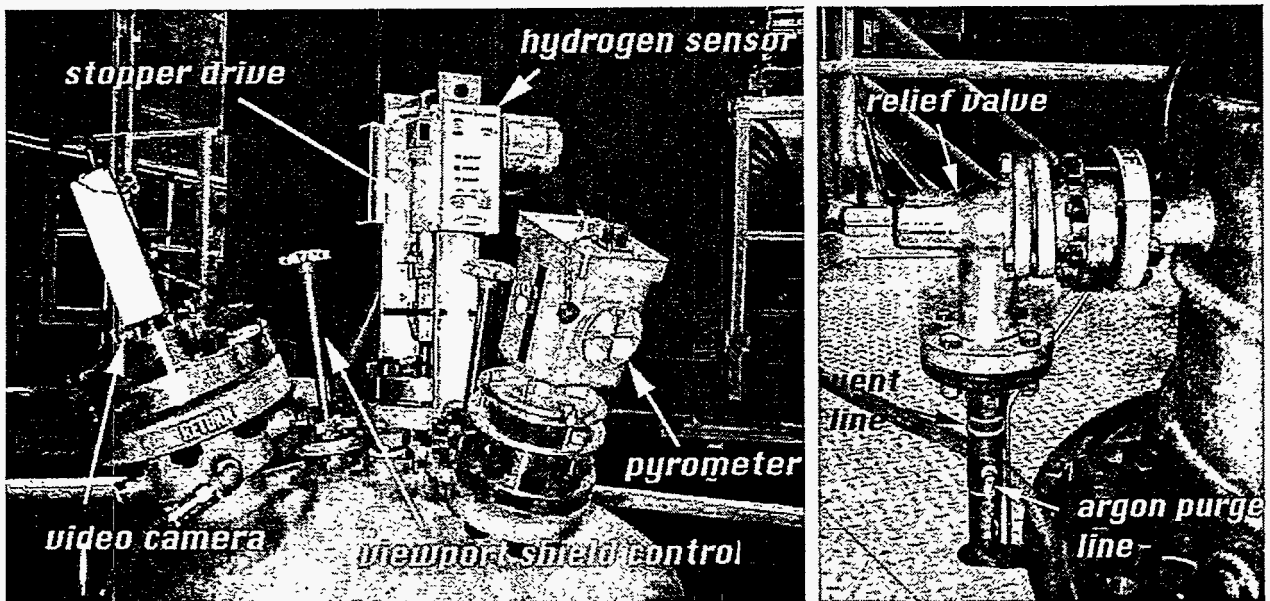


Figure 8. Left: Upper cover with 2 sightglasses and the stopper drive actuator port. Right: Pressure relief valve bolted to the body of the furnace. Argon bleed lines are used to dilute flammable mixtures in the gas supply and vent lines.

Overpressure protection is provided by a 38 mm (1.5 in.) diameter pressure relief valve model 2-600 set for release at 5.52 MPa (800 psi) and manufactured by Fluid Mechanics Valve Company (**Figure 8**). The overpressure line is vented outside the structure above the roofline.

The furnace is evacuated through a 38 mm (1.5 in.) pneumatically actuated Whitey ball valve (**Figure 9**). A 102 mm (4 in.) diameter tubing system connects the valve to a Sargent Welch model 1398 mechanical pump. The pump attains a low pressure level of 20 mTorr after pumping for 6 hours.

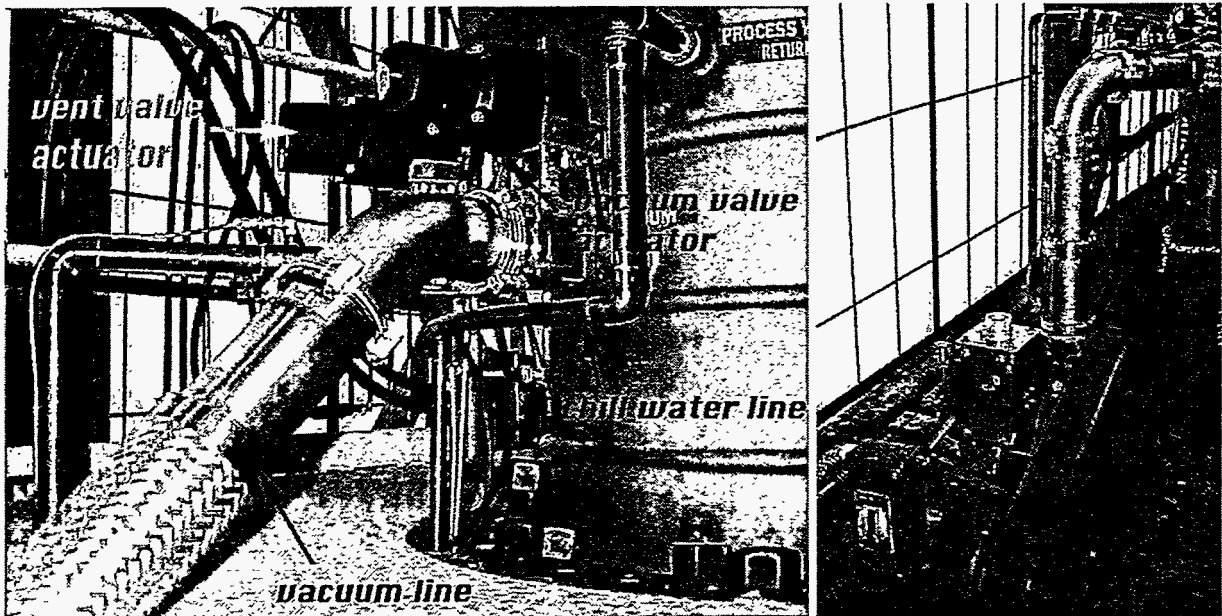


Figure 9. Left: Photograph of the vacuum line and Whitey 38 mm (1.5 in.) pneumatically actuated ball valve leading into the 100 mm (4 in.) OD flex vacuum line. Right: Photograph of the Sargent Welch vacuum pump and the 100 mm (4 in.) OD vacuum line.

The gas system (**Figure 10**) is made up of 3 manifolds that supply a single stainless steel 12.7 mm (0.5 in.) OD tube to the pneumatically actuated inlet Whitey 13 mm (0.5 in.) ball valve mounted on the furnace. Each manifold--nitrogen, argon and hydrogen--is supplied by three high pressure cylinders. The gas from each manifold passes through a check valve and a remotely controlled explosion proof valve as it is metered to the furnace supply line. The gas manifolds are located outdoors in a gas house.

A recirculating chill water system with large capacity solenoid valves supplies water to the furnace jackets and the casting mold chill plate. Five cm (2 in.) OD copper lines supply chill water to the system (**Figure 11**). Each sub-circuit is monitored with a flow switch preset to the minimum flow needed for that circuit.

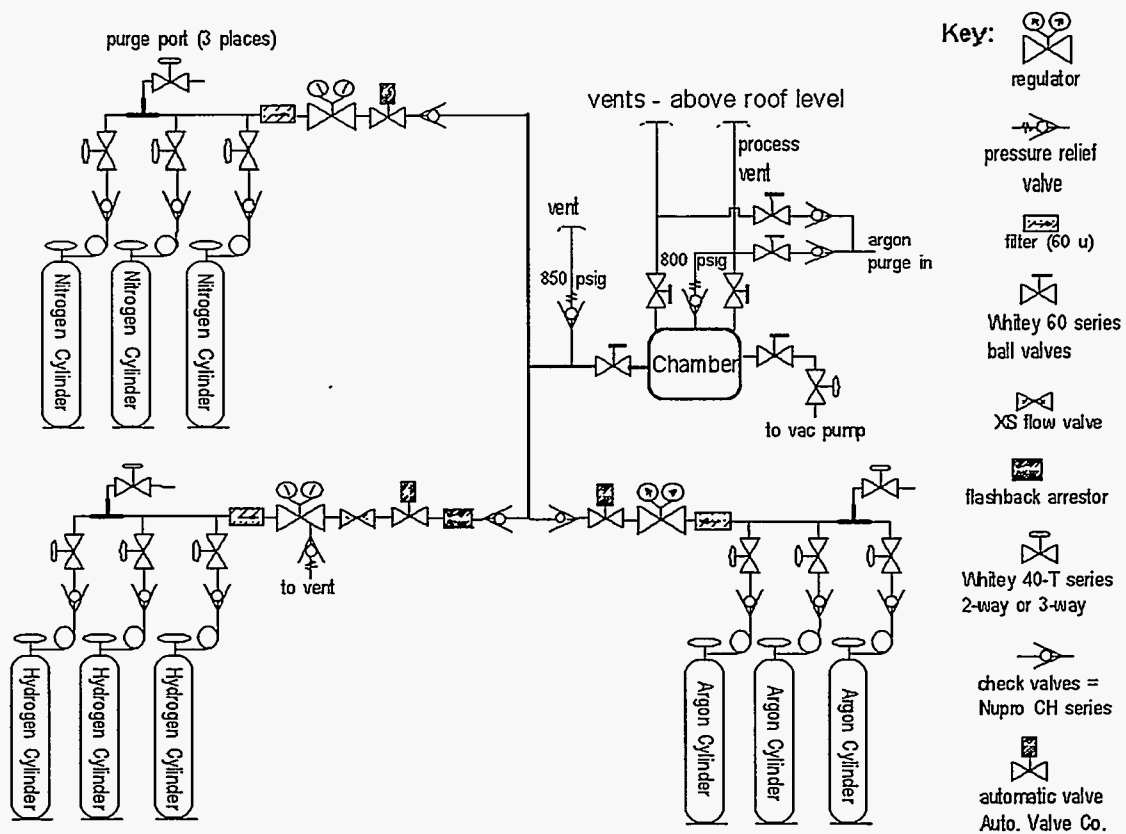


Figure 10. Schematic of the gas supply system.

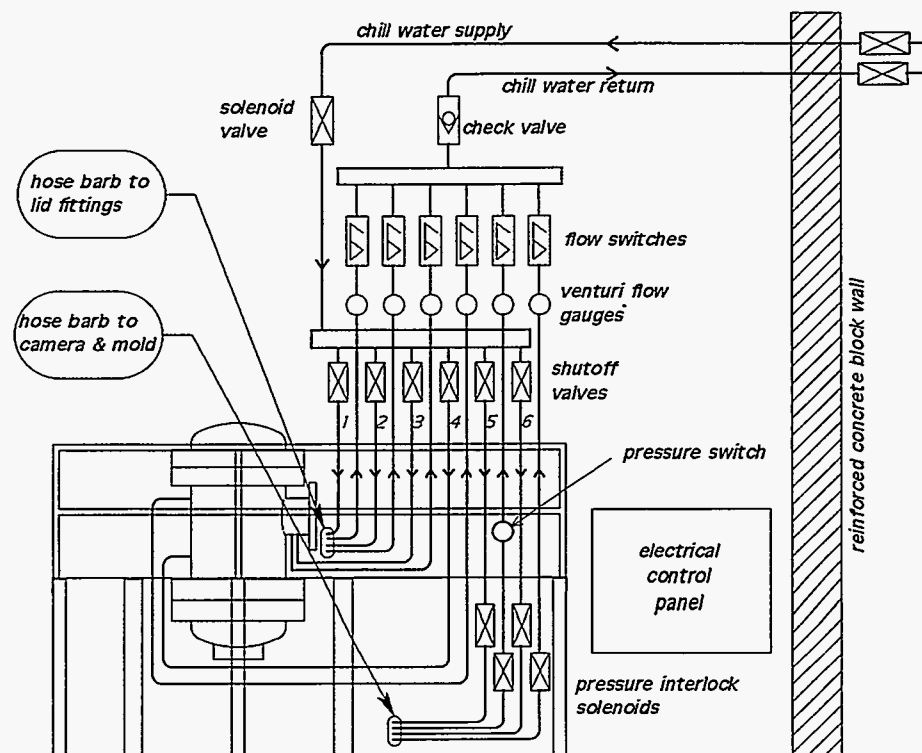


Figure 11. Schematic of the chill water manifold system.

Molten metal released from the Blasch crucible is poured through a tundish into a 25 cm (10 in.) ID ceramic ring atop a chilled copper plate. A second ring is placed outside and concentric to the first ring to contain spillage (**Figure 12**). The 29 mm (1.125 in.) thick copper chill plate has an integral spiral water path to allow the rapid heat transfer.

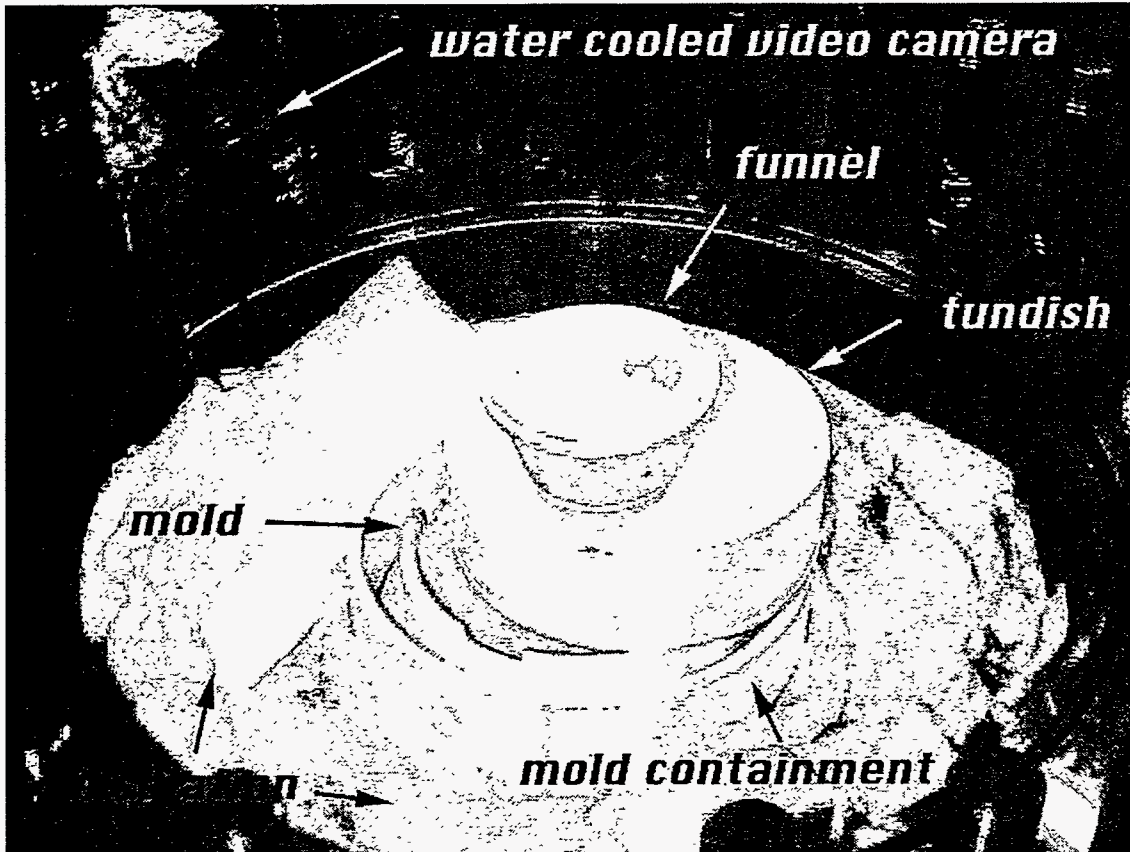


Figure 12. Photograph of the casting mold system.

Controls and Interlocks:

The controls for the Gasar system are located in the control room and in a control box station (**Figure 13**) next to the furnace.

The controls can be divided into three main systems: 1) the gas/evacuation system, 2) the stopper drive system and 3) the chill water system.

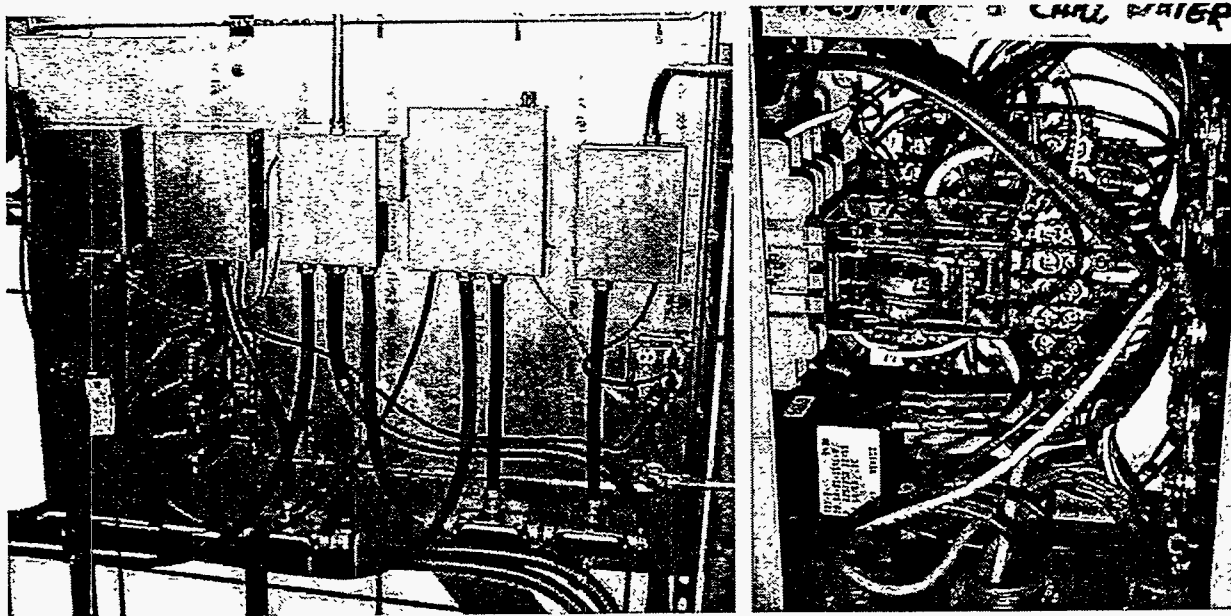


Figure 13. Left: Bank of control boxes next to the furnace. Five steel boxes are mounted on a large aluminum plate. Each box has a separate function. Solenoid valves introduce 90 psi air to four ball valves mounted on the furnace. Right: Typical internal components of a control box.

Each system has limit and interlock controls. Complete wiring diagrams are included in Appendices A-C. The control panel, mounted next to the induction power supply controls, is shown in **Figure 14**. Video screens are located in close proximity to the control panels in the control room. Data can be superimposed on the video screens with a system called "Video Panel Meter model 1400" manufactured by Outland Technology, Inc. The text is used to later identify information on specific melts.

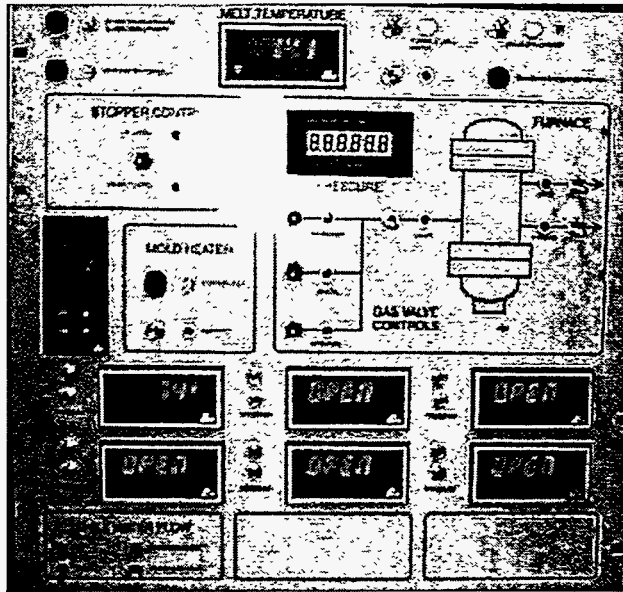


Figure 14. Photograph of the control panel. The main power switch to the 24 vDC systems is located in the upper left. The stopper control and indicator is on the left. Chill water controls are across the bottom and gas controls are on the center right.

Figure 15 is a photograph of the back of the control panel. A 24vDC supply provides power for some of the relays and controls. A 12vDC supply powers the Paroscientific, Inc. digital pressure signal conditioner and transducer. The pressure transducer is mounted on the control box station (**Figure 13**) and is connected to the furnace with a 6.35 mm (0.25 in.) OD stainless steel tube.

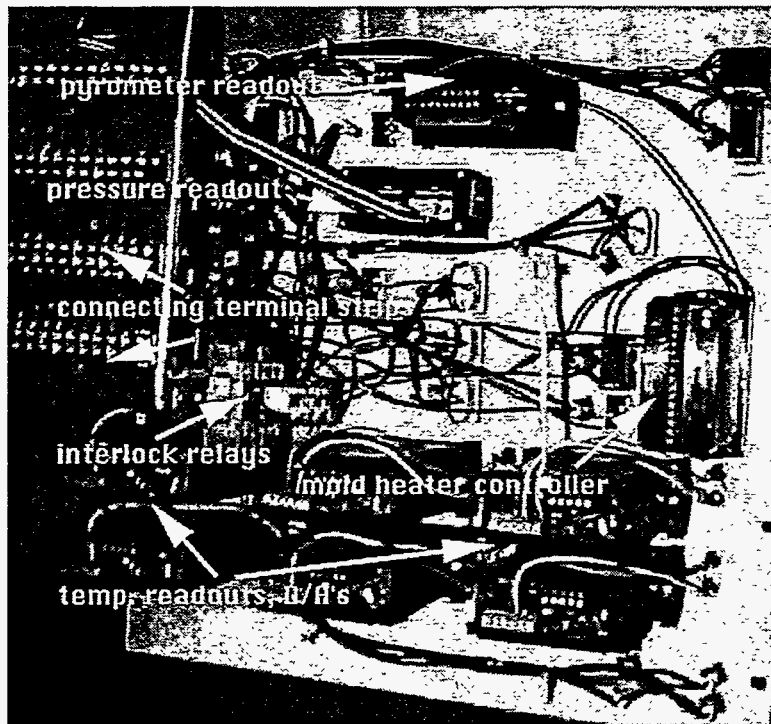


Figure 15. Photograph of the terminal strips, power supplies and wiring behind the control panel.

Gas Controls

The gas/vacuum system is separated into two sets of controls:

1. Three explosion proof 120 vAC solenoid valves on the gas manifolds allow H₂, N₂ or Ar to be supplied to one inlet valve at the furnace.
2. Four pneumatically actuated ball valves allow for venting, evacuation or introduction of gas. Solid state relays mounted in a box (**Figure 13**) energize solenoid valves which supply 0.62 MPa (90 psi) air to the pneumatic ball valves.

Figure 16 is a schematic drawing of one of the three gas control circuits. A SPDT momentary-or-constant-on switch on each system allows intermittent or continuous operation of each valve.

GAS CONTROL CIRCUIT

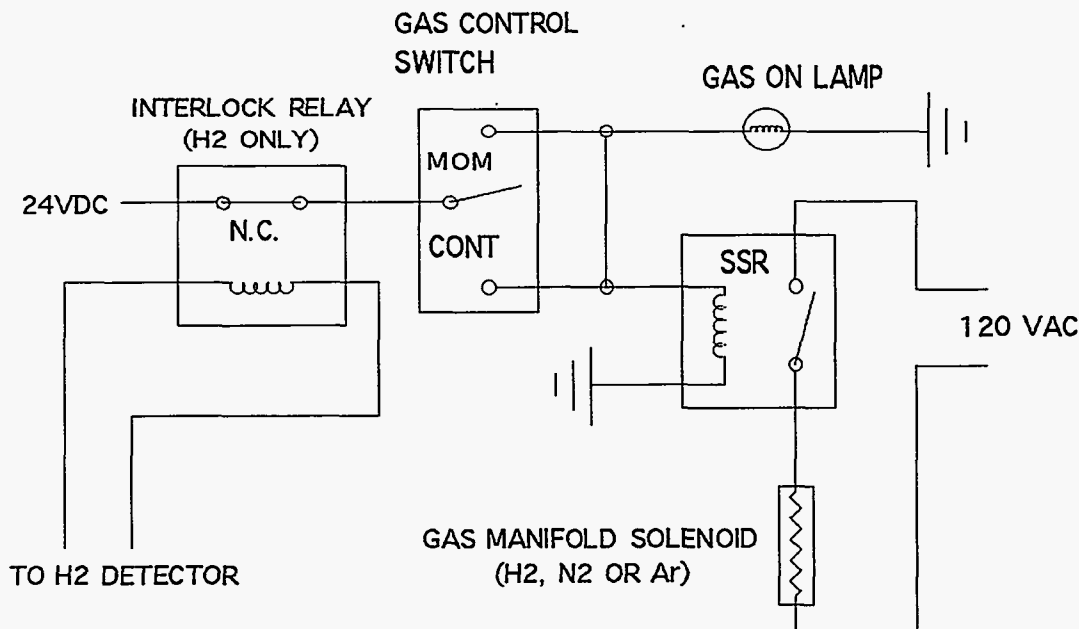


Figure 16. Typical schematic of gas valve manifold control circuit. If an H₂ detector trips power is cut to the SPDT switch on the H₂ circuit only.

The gas/vacuum system is interlocked in the following ways:

1. If either or both detectors are tripped, the H₂ manifold circuit is de-energized. One H₂ detector is mounted directly atop the furnace (**Figure 7&8**) and another is mounted above the furnace on the ceiling. The sensors, model HIC821, are manufactured by Industrial Test Equipment Co, Inc., and are designed to alarm at levels greater than 40ppm H₂.
2. The inlet and vent valve circuits are de-energized if the vacuum valve is open.
3. The vacuum or inlet valve cannot open if the furnace is pressurized to over 0.10 MPa (14.5 psig). A unique pressure sensor, manufactured by SOR Inc. (model 56BA-KB216-M4-C1A) is used in this circuit. The sensor has the ability to operate at pressure levels from high vacuum to over 10.3 MPa (1500 psi).

Stopper Drive Controls:

The stopper drive control system uses a Dayton 120 vAC reversible actuator to lift the stopper and allow molten metal to be cast into the mold below. **Figure 17** is a schematic of the system.

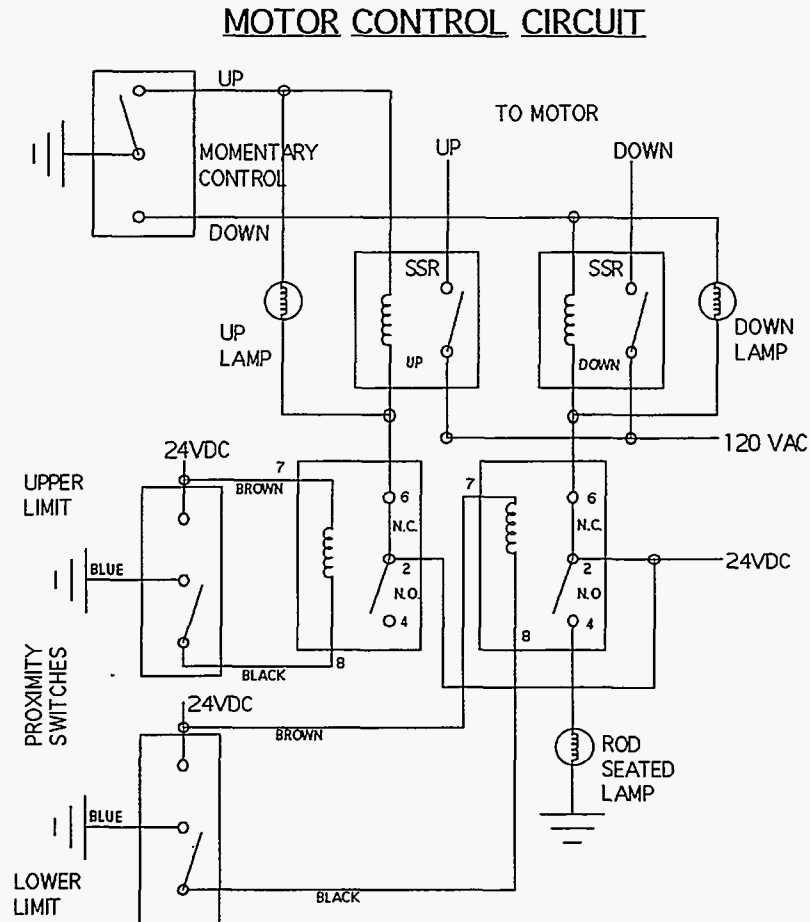


Figure 17. Motor drive electrical schematic.

Omron proximity switches are used to limit the travel of the actuator in both directions. When a limit switch is actuated, travel is allowed only in the opposite direction. Indicator lamps on the control panel illuminate when the motor is energized in either direction and when the lower limit switch is actuated.

Chill Water controls:

A recirculating chill water system supplies the Inductotherm power supply and the furnace in parallel. The power supply is internally interlocked to automatically shut down if flow conditions are not satisfied.

The furnace chill water distribution system, shown in **Figure 11**, further divides the large water supply into several circuits to the furnace jackets, mold chill plate and internal

camera. This system is monitored and interlocked with flow and pressure switches. The chill water solenoid valve allows on-off control of the flow into the manifold system. Other solenoid valves interrupt the mold and camera chill water circuits. The chill water valve is controlled with a solid state relay; the mold and camera chill water solenoids are controlled with a latching electromechanical relay. When flow to any of the monitored circuits is present, a lamp on the control panel is illuminated (shown schematically in Appendix A).

CHILL WATER OVERPRESSURE LATCHING CIRCUIT

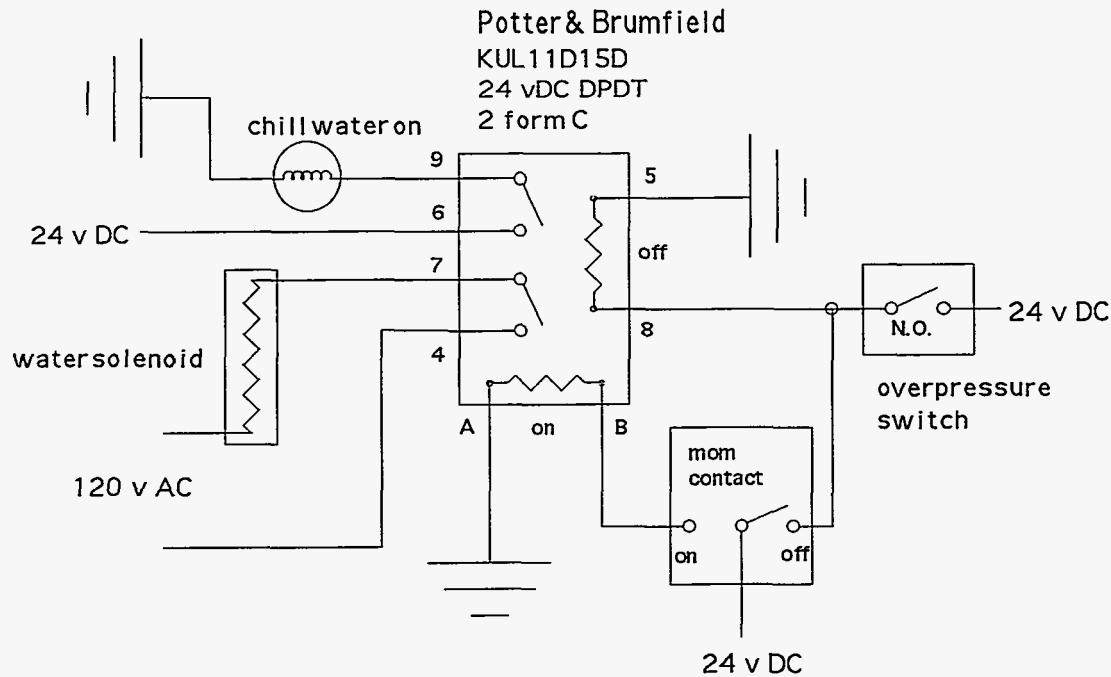


Figure 18. Latching circuit used to monitor overpressure of mold and camera chill water circuits. The chill water can be turned on only by activating the A-B coil in the latching relay. The chill water is turned off, thus isolating the furnace and preventing overpressurization of the rest of the chill water system, by activating the 5-8 coil. The 5-8 coil is energized by either the overpressure switch or the momentary on-off switch.

The mold heater/chill water system contains two interlocks—one that stops water flow to the mold and camera if the overpressure switch closes and one that turns off the mold heater if the flow is interrupted. The overpressure circuit, shown schematically in **Figure 18**, is used in the mold chill water system. A SPDT momentary action switch applies power to the latching circuit “on” or “off.” The overpressure switch completes only the circuit to the latching circuit “off,” thus providing the interlocking action. The mold heater circuit (appendix C) is energized through a solid state relay which can only be energized when the mold chill water flow circuit is completed. Three indicator lamps are used in the mold chill water/heater system. One is the “mold chill” indicator that is illuminated if the overpressure latching relay has gone to the “off” position, indicating an overpressure event has occurred. The second is the “mold flow” indicator which illuminates when water flow is present. The third is the “element” indicator which illuminates when the mold element switch is closed and water flow is present.

Run Procedure:

The following is a generic procedure for operating the porous metals furnace and was first described by Gutsch:

With the lower lid lowered and rolled out for easy access, the water chilled copper mold platen is coated with a uniform thin layer of investment casting prime coat slurry. This coating is a combination of tabular alumina and colloidal silica and is used to attach two concentric ceramic rings, the mold and the over-flow containment to the platen. A ceramic tundish and funnel system is placed atop these rings (**Figure 12**) in view of the internal camera. The tundish is located directly below the pour spout.

The charge is loaded into the crucible allowing sufficient clearance for the stopper rod. Once charged, the crucible lid is installed and the stopper rod sealed in its seat with ceramic slurry. The chamber is closed by raising the bottom flange and lowering the top flange into position. The o-ring flanges are sealed by tightening the nuts to the required torque value for the maximum anticipated pressure of the run. The stopper rod linkage, flange and rod actuator assembly are then installed.

Since the primary gas used is hydrogen, the furnace must be evacuated. A single mechanical pump evacuates the chamber to below 20 mTorr. If any of the gas supply system has been exposed to atmosphere it must be evacuated and purged using a gas driven venturi pump and backfill system. The main line to the inlet valve can also be purged in the same manner. Also, while roughing out the chamber, the chill water flow rates are set. The copper chill platen circuit is set to 19 liters/minute (5 gallons/minute).

After the pressure is reduced to < 20 mTorr, the heating process can begin. The charge is heated in vacuum until the system can no longer overcome the outgassing of the charge material, ceramics, etc. Above 750 mTorr, or when the charge has reached test temperature, the power supply is shut down and the chamber pressurized with the appropriate mixture of gases. After pressurization, the power supply is turned back on and the melt stabilized for five minutes to allow the gas to saturate the liquid metal.

The power is turned off and the melt is then cast into the mold where the gas-eutectic solidification process is completed.

A formal operating procedure exists in center 1800, SNL, OP #1897013.

Summary:

A melting/casting furnace has been built for the purpose of producing anisotropic porous metals. This furnace is capable of producing up to 17 Kg (37 lb.) of material in a melt, and was built to prove the viability of the process in industry. Initial research done by Gutsch, with this furnace, proved that Gasars can be successfully produced by applying the principles presented in United States Patent 5,181,549. The furnace controls successfully allow the manipulation of melt superheat, hydrogen partial pressure and total hydrostatic pressure for creation of multiple pore volume percentages and pore sizes.

References:

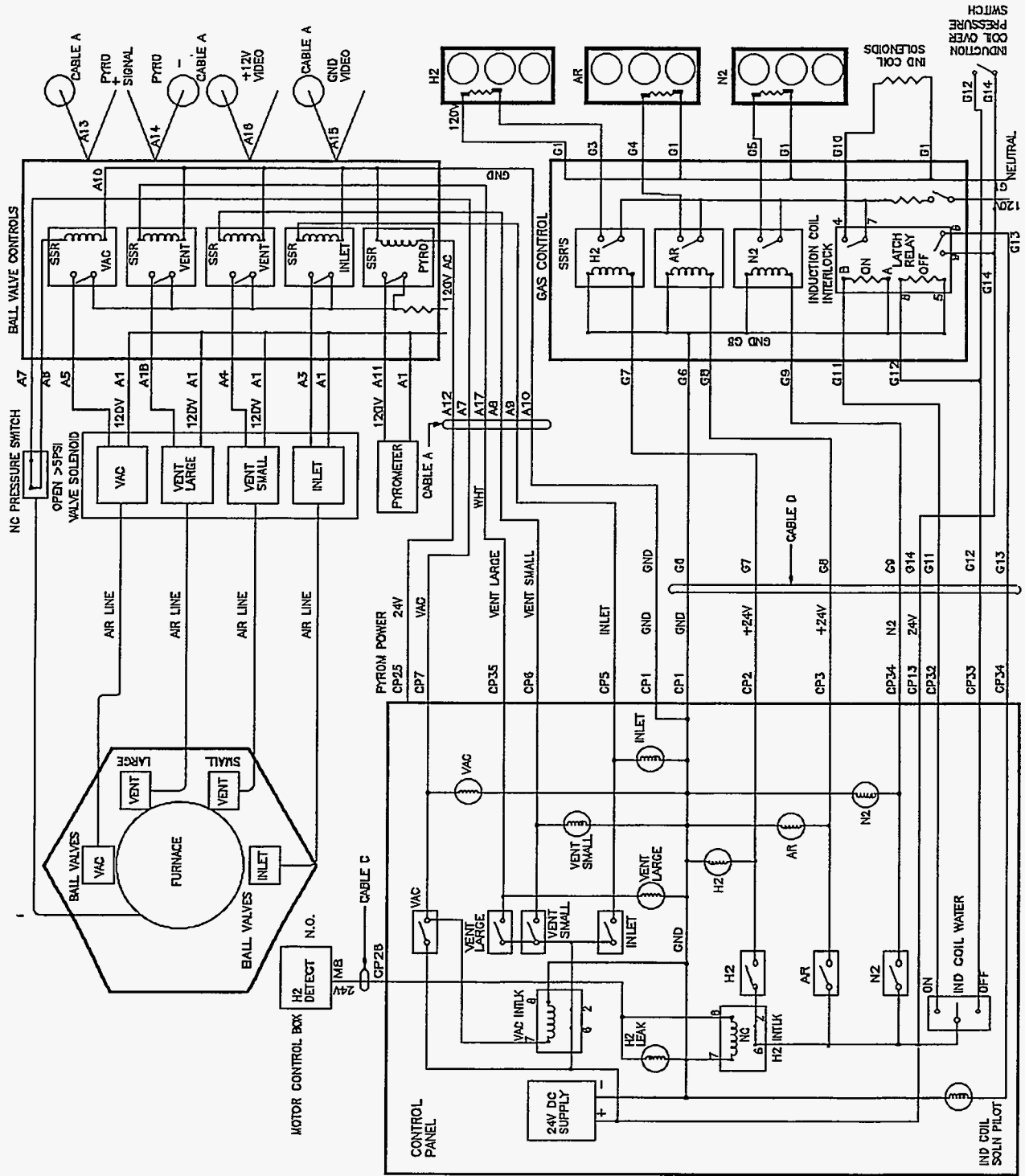
Baldwin, M. D. (1997). *Center 1800 OP For the Porous Metal Furnace at the Liquid Metal Processing Laboratory (LMPL)* Sandia National Laboratories.

Gutsch, T. C. (1998). *Production of Anisotropic Porous Nickel By Cast Metal Processing*, Masters' Thesis, CSU.

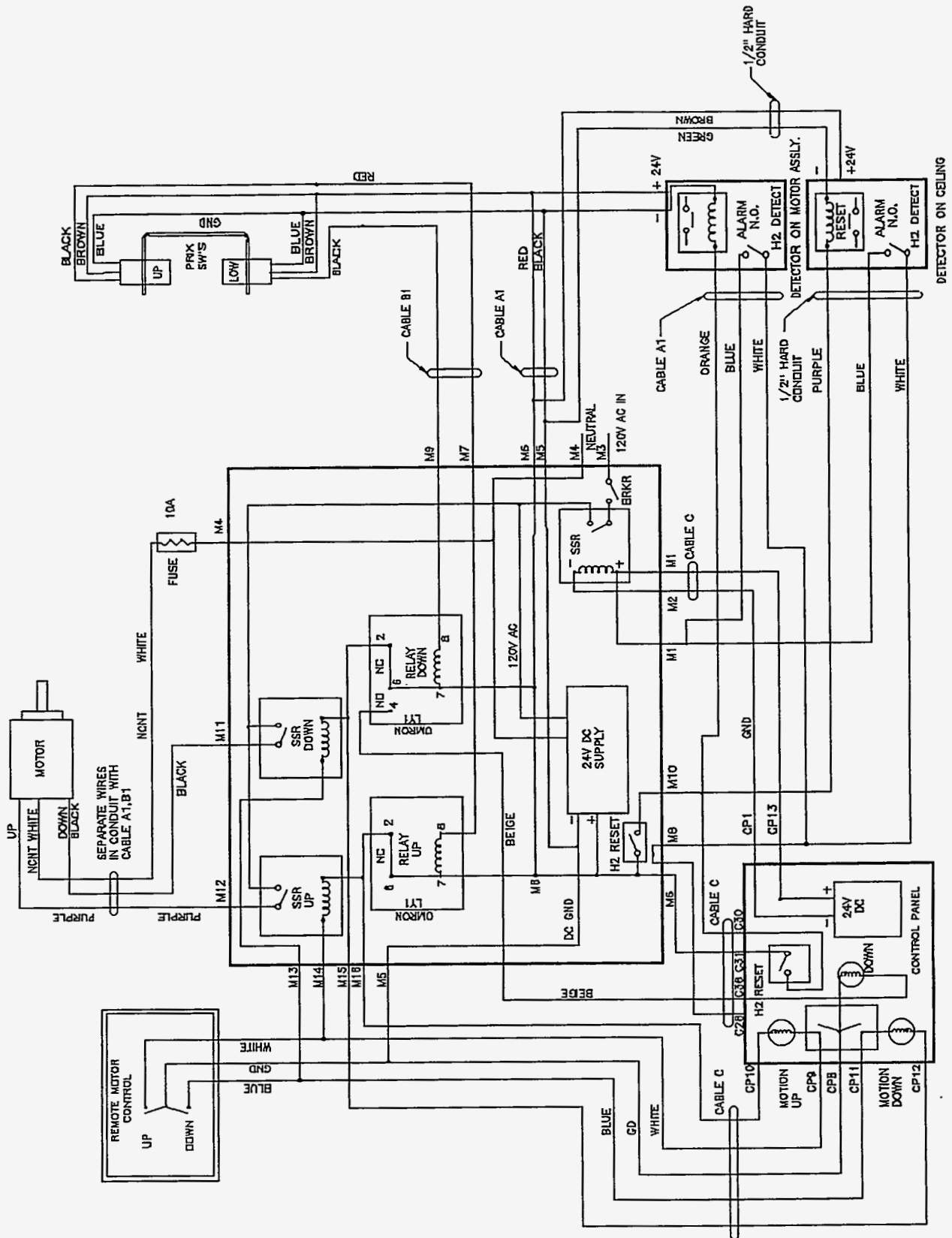
Shapovalov, V. I. (1993). *United States Patent Number 5,181,549 January 26, 1993.*

Walukas, D. M. (1992). *A novel approach in the fabrication of porous metals.* Unpublished manuscript.

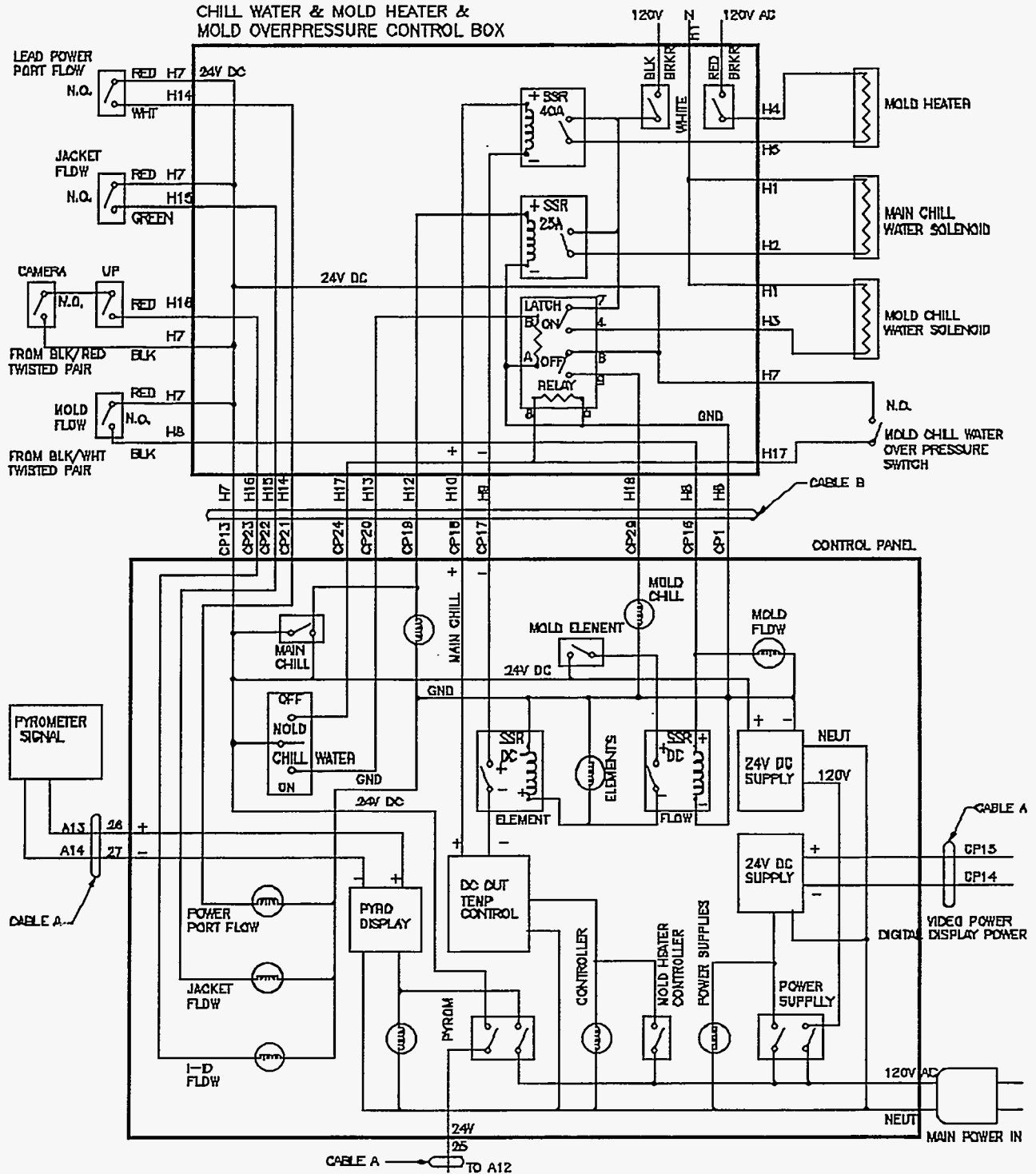
Appendix A: Gas control schematic



Appendix B: Motor control schematic



Appendix C: Chill water control schematic



Distribution:

1	MS	1435	K. Hayes, 1800
1		1411	D. B. Dimos, 1831
5		0340	D. T. Schmale, 1831
1		0333	F. G. Yost, 1841
1		1349	W. Hammetter, 1846
5		1134	M. Miskiel, 1846
1		1134	M. Baldwin, 1846
2		0563	J. Padilla, 1486
1		0873	R. Ney, 14402
1	MS	9018	Central Technical Files, 8940-2
2		0899	Technical Library, 4916
1		0619	Review and Approval Desk, 00111 For DOE/OSTI
5			Thomas Gutsch, Thermal Technology, Inc. 1911 Airport Blvd. Santa Rosa, Ca. 95403