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Development of a Vacuum Hot Pressing
Apparatus for the Production of
Metal Matrix Composites

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DEVELOPMENT OF A VACUUM HOT PRESSING APPARATUS

FOR PRODUCTION OF

METAL MATRIX COMPOSITES

By

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ABSTRACT

DEVELOPMENT OF A VACUUM HOT PRESSING APPARATUS FOR PRODUCTION OF METAL MATRIX COMPOSITES

By

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A vacuum hot pressing apparatus that was developed for the production of metal matrix composites is described. Characteristics of, and operating instructions for the vacuum, furnace, press, and data acquisition systems are included.

Preliminary specimens of metallic glass ribbon reinforced, metal matrix composites were produced and are briefly characterized.

The performance of the device in these initial experiments was as designed, with vacuum, furnace, and press action well within the required operating range.

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Section 1. Introduction

This project was undertaken with the intention of developing a vacuum hot pressing apparatus for the production of metal matrix composite materials. Specifically, it was intended to replicate the work of S.J.Cytron[1], who produced composites consisting of metallic glass ribbons supported by a matrix of a superplastic aluminum alloy, and to extend these researches to other metallic glass/metal matrix systems.

This process presents a number of experimental difficulties, principally, the metallic glass must be kept below its crystallization temperature while the matrix material must be hot enough to ensure superplastic flow. The temperature distribution within the hot pressing die must, therefore, be closely controlled and monitored. Also the pressure within the die, the vacuum within the chamber, and the ram travel must all be measurable in order to obtain useful experimental data. Merely squeezing samples without knowledge of the conditions they are subjected to would be useless.

Interest in these materials stems from the extremely high strengths and toughnesses that some metallic glasses have displayed, from their potential for low cost volume production, and from the fact that utilization of these properties requires that they be incorporated into a composite.

that a flexible and adaptable system would be obtained, and at a substantially lower cost than that for purchasing a commercial device.

The primary intention of this thesis is to provide a concise description of this vacuum hot pressing system, which includes its basic operational characteristics and instructions for the benefit of those who may employ it in future research at MSU.

Since the system was designed with flexibility and modifications in mind, its characteristics may change substantially from those described. Up to date information must therefore augment this thesis.

Section 2. Vacuum Hot Pressing System Overview

2.1 System Description

The system consists of a vacuum chamber with an internal three zone electric furnace, and an externally mounted screw driven press which transmits force with a ram that passes through a linear motion feedthrough to a die within the furnace (figures 1 to 5). Thermocouples are provided to monitor the temperature within the furnace and die, and a compression load cell is linked to a second ram to measure die pressures. Vacuum is obtained with a high capacity diffusion pump and mechanical roughing and backing pump, and monitored with a compression manometer (Mcleod gage) and a cold cathode ionization gage.

2.2 Performance Specifications

The maximum attainable loads are limited by the thrust bearing in the screw advance mechanism (section 3.1). The working loads of bearings are related to the speed at which they operate, with higher speeds leading to lower permissible loads, and to the service life of the bearing. In this case the loading is essentially static, and the service life fairly short, so that the loading can be much higher than that for dynamic, long-life applications. By consideration of these factors and from data for similar types of bearings, a maximum loading of 2,000 kg may be estimated.[2] Higher loads may be tolerated briefly, but will shorten the bearing life and could cause catastrophic failure.

Section 3. Press System

3.1. Advance Screw and Gear Reduction System

The advance screw and drive system were obtained from a Scott Testers Inc. tensile testing machine. Originally intended for use in tension, this system consists of a 6 turns per inch, 1.125 inch (2.8575) diameter, acme thread screw which is driven up and down by a combination nut which is support by a thrust bearing. This nut is driven by a beveled gear through a gear reduction system. Unfortunately, this gear box is equipped with a ratchet - clutch - forward to reverse gear shifting mechanism, which somewhat complicates operation. When in drive mode, an inner sliding gear system is moved to the right, and the ratchet mechanism will engage and solidly advance the screw. When in retract mode, the inner sliding gear will be shifted to the left and will disengage and not advance the screw. In this mode the gear box is in reverse, and the screw will try to retract. This retraction force is limited by a clutch however, so that if the ram is tightly wedged, it will be necessary to release it be loosening the load cell crosshead nuts on the main press columns.

Shifting the gear box back to drive mode is accomplished by pulling on the lever beneath the box.

The smaller, side-mounted gear box reduces the input RPM from the servo motor and pulley system. One revolution at the input of this gear box will advance the screw 1/240 of an inch. (.1058 mm).

3.2. Press Frame

The press frame was modified from the original tensile testing apparatus to increase stiffness and to support the chamber. Nevertheless, it should be regarded as a "soft" frame, that is, it will deform somewhat when loads are applied so that the displacements achieved within the dies will not exactly equal the displacement of the screw. For this reason accurate values of the ram travel must be obtained by directly comparing the positions of the upper and lower rams by an LVDT (linear variable differential transformer) or other device, rather than by inference from the screw motion.

The press rams themselves are supported and guided independently from the screw advance mechanism to simplify press alignment. Thus, the lower ram is aligned by the linear motion feedthrough and a lower alignment sleeve, with the two being supported by an aluminum yoke. The flange that forms the top half of this yoke, bearing the linear feedthrough sleeve, was positioned with respect to the lower half by inserting the lower ram and ensuring its freedom of vertical motion, and pinning the aluminum flange accurately in place and bolting it down. The linkage between the lower ram and the advance screw

has four degrees of freedom for small displacements, allowing slight lateral shifts and tilts to reduce the stringency of the required alignment. The top surface of the advance screw is slightly convex, directly contacting the flat lower surface of the lower ram, but allowing these motions.

The top ram is aligned by the upper linear motion feedthrough sleeve and by contact with the die and the load cell. This minimal support is intended to reduce frictional drag that might introduce errors in load measurement. The surface that contacts the load cell loading ball is a 120° concave cone of the same geometry as the original Instron load cell shaft.

3.3. DC Servo-Motor Drive

The DC servo-motor is an Electro-Craft torque motor with an output torque approximately proportional to the applied current. With a rated maximum voltage of 12.0 volts and maximum current of 11.9 amps, the servo motor produces about 6.5 ounce inches of torque per ampere up to a maximum of 70 oz. in. (5038 gm. cm). Additional data may be obtained from the motor's engineering specifications sheet.

Power to the motor is provided by a circuit (figure 6) which produces a DC output with a voltage adjustable between 0 and 12 volts with a 20 ampere rating. This is obtained by using a variac to provide 0 to 120 VAC, continuously adjustable, and reducing this voltage with a step down

transformer and rectifying and filtering it.

The potential for this motor can be controlled by feedback from the load cell for a kind of limited servo-controlled action. However, the latching effect of the advance screw makes this a one-way control only, the load cannot be lightened under automatic control, but only increased or maintained.

The linkage between this servo motor's output and the advance screw gear box input may be changed to provide different maximum press loads, with greater gear reduction ratios leading to higher maximum loads. Belt drives with different ratios and two gear reducers with 10/1 and 15/1 ratios are provided for this purpose, however extreme reductions should be avoided so that the stalling torque of the motor is reached before destruction of the advance screw thrust bearing.

3.4. AC Synchro-Servo Selsyn Motor Drive

Although not yet implemented, AC synchro-servo selsyn drive motors are also available to provide press advance at a controlled, constant rate. Since the press frame is "soft" (section 3.2) this will not lead to an exactly uniform ram motion but only to an approximation of it. Pairs of these motors operate synchronously, one, the "generator", being driven by a gear reduction motor while the second, the "servo follower" follows its rotation in exact synchronism. This second motor will be linked to the screw advance system by a gear and pulley system to drive the screw advance mechanism.

Section 4. Furnace System

4.1 Furnace

The furnace consists of three sets of nichrome heating elements supported within a type 303 stainless steel housing by ceramic insulators in a split-cylindrical geometry (figure 7), and insulated by a layer of quartz wool wthin an outer stainless steel sheath. The furnace is divided into 3 zones, a 7.5 centimeter high central zone and 3.75 centimeter high upper and lower zones, with each zone controlled by the ATS controller.

The inside diameter of the furnace is as large as possible while leaving clearance between the press columns. This allows for flexibility for future applications and tends to smooth out any irregularities in the temperature distribution, that is, the die is more uniformly heated than would be the case if the die-furnace clearance were made smaller.

Transfer of heat from the elements to the die is almost entirely by black body radiation. The equation for this transfer for the case of a small convex object to or from a larger, continuous enclosure is given by Duffie and Beckman [4] as:

$$Q_1 = \varepsilon_1 A_1 \sigma (T_2^4 - T_1^4)$$

where: Q_1 = heat transfered, ε_1 = emissivity of the object

σ = Stefan - Boltzmann constant (5.67×10^{-8} W/m² K⁴

and T_1 and T_2 are the temperatures of the body and enclosure in degrees Kelvin

This equation may be used as a guide in estimating heat transfer rates in the furnace, however, the actual geometry of the heating elements, die, and furnace walls differ from this case and so the actual conditions will be somewhat more complex.

4.2. Controller

The furnace controller is an Applied Test Systems series 2010 programmable microprocessor controller. Detailed specifications and operating instructions may be found within its manual.[4] The controller is set up to operate a central furnace zone, based on feedback from a thermocouple, and two end zones. These end zones are not independent but are linked to the central zone with variable offset adjustments to allow for maintaining different temperatures or compensate for different heat loss rates.

The ATS controller produces three - 220 VAC outputs rated at 10 amperes each which are switched by silicon controlled rectifiers to produce a variable output by adjusting the point in each half cycle, at which the rectifiers are made conductive. Since these are latching, avalanche type devices, they continue to conduct for the rest of the half-cycle, or until the current through them goes to zero.

4.3. Voltage Reducer

Since the 220 volt output of the controller is a high

enough potential to cause corona discharges at the low pressures present within the chamber, a voltage reducer consisting of a single 20 amp continuously variable autotransformer, and two 12 amp autotransformers which are set up to provide fixed voltage outputs (figure 8), has been added. Since the current capacities of these units exceed that of the controller, and for other safety reasons, a three pole, 10 ampere circuit breaker is interposed between the controller and the voltage reducer. This breaker will interupt all three lines in the event that any line exceeds the 10 ampere rating of the SCRs in the controller, preventing burnout of these and other components. This represents a significant change over the controller's internal, single, 30 amp breaker which will allow each 10 amp zone to burn out in turn, provided that the total amperage does not exceed 30. This added safety system should not, therefore, be bypassed.

The voltage outputs from the reducer should not exceed 100 volts due to the previously mentioned corona discharges. Higher temperatures are achieved by the controller increasing the percentage of "on" time, rather than by adjusting this voltage. The high potential wattage output of the furnace's low wattage density heating elements makes this possible.

4.4 Temperature Measurement

Die and furnace temperature measurement is accomplished by means of stainless steel sheathed thermocouples which pass

through feedthroughs described in section 5.6. The thermocouples employed may be of different types provided that they have the appropriate temperature range and that the thermocouple monitor is of the same calibration. The Omega digital thermocouple thermometer has a calibration switch behind its front panel for matching the different types.

The proper location of the thermocouple probe tips is essential to yield meaningful temperature measurements, as well as for establishing temperature uniformity and detecting gradients that may be present.

Section 5. Vacuum Chamber and Feedthroughs

5.1 Chamber

The vacuum chamber was obtained from a National 3640 anaerobic incubator. Its interior measures 30.5 cm X 30.5 cm X 48.26 cm with a wall thickness of .476 cm. The flat walls facilitate mounting of feedthroughs and other components. The front entry/window is Pyrex glass, 1.25 cm thick. Elastomer gaskets provide seals between this glass and a metal frame, and between the frame and the front edge of the chamber. This latter gasket is removable and should be periodically inspected for deterioration, cracks, and other damage. A thin film of silicone vacuum grease should be maintained on this seal, which could prevent high vacuum operation if not in good condition.

The chamber material is a non-stainless steel and should therefore be protected from contamination by corrosive agents and humidity. Rust is a hygroscopic material and would outgas extensively if allowed to form on the chamber interior. For this reason the chamber should left in an evacuated condition following use.

5.2 Linear Motion Feedthroughs

The linear motion feedthroughs are of a standard design taken from A. Roth[8],(pg. 512), (figure 9). The use of dual o-rings improves the overall performance and allows for the possibility of pumping the space between the two seals down to a moderate vacuum. This procedure greatly improves sealing performance in these seals, since only a very small pressure differential then exists to force gas past the inner seal. Since the seals appear to be performing adequately without this provision, it has not yet been implemented.

5.3 Column Feedthroughs

The seals between the coarsely threaded columns and the relatively thin chamber walls are of a slightly unusual type (figure 10). The seals were produced by modifying flange nuts with a sufficiently wide and thick flange by machining an o-ring retaining groove in the base of the flange to seal against the chamber wall, and by machining away the threads on a portion of the nuts and of the columns to form a second o-ring retaining gland. Removal or adjustment of these flange nuts should be minimized to avoid damage to the o-rings,

5.4 Electrical Feedthroughs

The electrical feedthroughs are modified 1/4 inch compression fittings which have flanges and nuts designed for passage through a wall. The internal diameter has been drilled out to provide 1/4 inch clearance through the feedthrough length. Brass rod 5/32 inch in diameter is passed through Tygon tubing for electrical insulation which in turn is passed through the compression fitting. The compression ferrules and compression nuts are passed over the tubing and tightened and the whole assembly is passed through the chamber walls and held in place by the flange and nut.

Silicone vacuum grease is liberally applied between the rod and tubing, to the compression ferrules, and to the seal between the flange and the chamber wall to improve sealing. Since Tygon tubing will outgas in a vacuum, the exposed length of tubing in the chamber interior must be minimized and should be coated with silicone grease. The feedthroughs should be checked for ohmic heating at high furnace temperatures since the Tygon will have a high vapor pressure at elevated temperatures and will soften and decompose. If this becomes a system limitation the brass rods may have to be replaced by a water-cooled copper tubing arrangement, similar to a heat pipe. Similarly, replacement of the Tygon by higher temperature, lower vapor pressure plastic is desirable.

Within the chamber the brass rods from the feedthroughs are linked to the furnace through braided copper connection wires which have high surface areas to maximize heat loss through radiation. It is important to bear in mind that the normal condution and convection cooling modes of electrical wires are lost in a vacuum, and so all connections must be made with oversized wires, with large surface areas if possible.

5.5 Thermocouple Feedthroughs

The thermocouple feedthroughs are of the same design as the electrical feedthroughs, with the exception that the thermocouple sheaths are slightly smaller (1/8 inch) in diameter than the brass rod stock. This leaves the Tygon tubing slightly less compressed and necessitates more care in maintaining a proper seal and avoiding disruption of the seal by motion of the thermocouple after insertion.

5.6 Coolant Feedthroughs

The coolant feedthroughs for the rams were retained from the original vacuum chamber configuration and consist of 1/4" compression fittings with their interior drilled out to 1/4" i.d. and which have been permanently brazed to the chamber walls. Since compression fittings operate by radially forcing a ferrule into contact with a tube by axial compression, these must be securely tightened to ensure good contact and should also be heavily greased. Out-of-round tubing makes this type of seal very difficult and care should be taken to use good condition tubing and avoid any bending or other deformation in the event that the coolant tubes are replaced. Similarly, care should be taken to avoid bending or otherwise applying stresses to the mounted coolant tubes, since this will destroy the seal.

5.7 Diffusion Pump Mounting Bolts

The eight bolts that secure the diffusion pump to the chamber pass directly through the chamber walls, rather than through a seperate mounting flange that is not under a vacuum, as is the usual attachment method. This neccesitates the use of special o-ring sealing washers. These washers form axial o-ring seals with the chamber wall from the interior, and combined radial and axial seals with the top portion of the bolt shaft and the base of the bolt head respectively.

5.8 Leak Detection

The attainable vacuum is limited by gases leaked through the numerous fittings and seals, by outgassing from the chamber walls and contents, and by back streaming from the diffusion pump itself. Leaking and outgassing in particular must be minimized for adequate operating vacuum levels. A leak corresponding to 1 cc/second at atmospheric pressure would require a pumping system with a 10,000 liter/second capacity to maintain a vacuum in the range of 10^{-4} torr.

Comparison of the vacuum gage reading before and after tightening one of the compression nuts or otherwise tampering with any of the other seals is a means of indicating whether a leak may be present. Any substantial change implies that the seal may be faulty and should be dismantled and repacked with grease or otherwise improved.

Instructions for another method of leak detection may be found in section 6.5.

Section 6. Vacuum Pumping System

6.1 Vacuum Pumping System Overview

The vacuum pumping system consists of a TM - 641 6" diffusion pump with a pumping speed of 1200 liters/second (see graph), and Precision Scientific Co. PS - 300, two-stage oil-seal roughing and backing pump with a 300 liter/minute capacity. The system is a "valveless" design, that is the diffusion pump is directly connected to the chamber without an intervening valve. Valves are provided between the An optically opaque baffle that is integral to the diffusion pump and a second baffle within the chamber that also helps prevent objects from falling into the pump inlet.

6.2 Diffusion Pump

The TM-641 diffusion pump has an unusual heater design that allows very rapid heating and cooling, reaching full pumping speed from a cold start in about 5 minutes and cooling in a similar amount of time. This heating element consists of a fiberglas wick which vaporizes the diffusion pump oil in contact with it very rapidly, while the remaining pool of oil is at a lower temperature.

The base plate of the pump is cooled by circulating water, although the design for this is very poor. The water path is formed between the stainless steel bottom of the pump housing and an aluminum plate with a machined channel, the two being

sealed by a pair of concentric o-rings. The choice of disimilar metals has led to extensive corrosion, and to degradation of the o-ring seals, although this results only in slight water leakage from the pump and does not jeopardize the vacuum integrity.

6.3 Roughing/Backing Pump

The roughing and backing pump is a standard two-stage oil-seal mechanical pump with a 300 liter/minute capacity (Precision Scientific model PS - 300). This pump has a 1 horsepower motor which appears to be slightly overloaded and so should be fan cooled to minimize the temperature rise.

The pump is equipped with a gas ballast valve located at the top. If quantities of humid air are pumped, moisture may collect within the pump and this valve should then be slightly opened to flush more air through the second pump stage and eliiminate this condensation. This condition should not arise unless extensive rough pumping is done in humid weather.

The pump oil employed is CVC 70/19 with a vapor pressure of 1×10^{-5} torr. The pump was cleaned with CVC pump flushing fluid prior to filling.

6.4 Valves, Connectors, and Tubulation

All valves and tubes employed in the backing connection are as large in diameter and as short as possible to maximize conductance and provide tolerance for bursts of gas from the diffusion pump without exceeding its 450 micron maximum backing pressure. The placement of the roughing and backing valves is based on this criterion, rather than on operator convenience.

The valves and fittings employed are based on the Leybold-Hereaus KF series connectors, KF - 40, 40 millimeter i.d. for the backing, and KF - 25, 25 millimeter i.d. for the roughing valve and connectors. The roughing and backing valves are right angle, o-ring sealing types, and are employed to obtain a rotational degree of freedom and minimzed the number of components in the system.

The mechanical pump is vibration isolated by a KF - 40 stainless steel bellows tube which also allows freedom of motion for the pump in relation to the other components, and by padded supports for its mounting bracket.

6.5 Vacuum measurement

Measurement of the vacuum is achieved by a Mcleod gage (compression manometer) and by a Penning gage, and cold-cathode ionization gage, which are mounted to the roughing connection at the cop of the chamber.

The Mcleod gage operates in the range from 1 to 5,000 torr and functions by compressing a sample of gas and comparing its pressure to the uncompressed vacuum with a manometer arrangement. Condensible gases will cause this gage to give an erroneous reading, but aside from this difficulty, these gages are reference standards and should provide high accuracy within this range. Operation proceeds by allowing the gage to be pumped out while it is in the horizontal position, then tipping the gage up on end to seal off the bulb, compress the gas within it, and obtain the reading. The reference column of mercury should be aligned with the 0 level indicated, by slight tilting, if necessary. Additional instructions are printed on the back of the gage itself and in an instruction booklet provided with it.

The ion gage operates by ionizing gases within the vacuum by a high voltage potential, magnetically deflecting these ions to cause them to strike other gas molecules and ionize them in turn, and measuring the current that flow through the potential as a result. This gives a gage reading that is somewhat dependent on the nature of the residual chamber gases. This effect may be exploited for leak detection by the following means. Butane gas may be used to flood the area outside the chamber near the expected leak (CAUTION: butane is explosive). If there is a leak present the butane will pass through and give a different reading than that from air passing through the same leak, thereby indicating its presence.

Section 7. Data Acquisition System

7.1 Load Cell

The load cell is an Instron compression type with normal full range loads of 200, 500, 1,000, 5,000, and 10,000 lbs, (≈90 - 4500 Kg). The original loading shaft has been replaced by the upper ram, which has a concave 120° conical surface that directly contacts the loading bearing. The load cell is mounted on the crosshead of the Carver laboratory press by brass spacers with 3/8 inch N.C. threads to leave clearance for the electrical cables. Excitation and output monitoring of the load cell should be performed according to standards set in the Instron manual.

7.2 Thermocouple Probes

Temperature measurement should proceed according to the procedures outlined in 4.4. Care should be exercised to ensure that the thermocouples employed are of the same type as the calibration setting of the meter or controller employed. Several measuring point are desirable to provide a clear picture of the variation of temperature with time and position within the furnace and die. Most thermocouple meters have recorder outputs which provide a standard signal for the A/D converter of chart recorder. Prior calibration of the recorded output is desirable, although the meters themselves should provide accurate indications without this, provided that connecting wires are of the same thermocouple alloy, or that the junctions of copper wires, if used, are maintained at the same (room) temperatures.

7.3 Vacuum Gages

Vacuum monitoring should be performed according the the guidelines in 6.5. Since the ion gage does not supply a recorder output, the vacuum levels maintained during a press cycle must be manually recorded at this time. The addition of such an output to the gage is being explored.

Section 8. Operating Instructions

8.1 System Loading and Preparation

Assemble the specim to be hot pressed atop one of the pistons, including whatever spacers or seperators may be appropriate and load the piston into the die assembly (figure 11). Insert the upper piston and the upper and lower insulators into the die and set the die in place atop the lower ram, allowing the ram to just pass into the graphite die and leaving clearance in the die for the upper ram. Raise the ram with the ram advance mechanism at a slow speed until the die and specimen are lightly clamped in place.

Install the front furnace half and attach the electrical conducters. System pumpdown may then proceed.

8.2 Vacuum System Operation

With the sample loaded and the die and furnace preparations completed;

Start-up procedure:

1. CLOSE vent valve
2. CLOSE backing valve
3. CLOSE roughing valve
4. Switch backing pump ON
5. Hold chamber door closed while OPENing roughing valve

Note: The "smoke" that is produced by the mechanical pump at this stage is a normal consequence of its operation.

6. After about five minutes, check the vacuum attained with the Mcleod gage, it should be in the range of 10 - 50 microns (.01 - .05 torr).

7. Switch ion gage ON, set range switch to ".03"

8. When the vacuum has reached about .01 torr, as indicated by the ion gage, OPEN the diffusion pump water cooling valve enough to maintain a steady flow rate of about 2-4 liters/minute and check to ensure that water is in fact flowing.

9. Turn diffusion pump ON

10. Monitor ion gage. The pressure should increase slightly to about .03 torr for a few minutes and then decrease rapidly as the diffusion pump comes up to speed after about five minutes.

11. Check the temperature of the diffusion pump cooling water at the drain. If it is appreciably warmer than tap water, increase the flow rate.

12. After about 10 minutes, compare the vacuum achieved with that usually obtained to check for leaks. It should be at least as good as 10^{-4} torr.

13. The system is now evacuated and ready for heating.

14. Follow the shutdown procedures outlined below following hot pressing.

8.3 Furnace Operation

With the specimen loading completed and the chamber evacuated, set the desired temperature on the ATS controller, switch the selector to vacuum hot press and switch the circuit breaker ON. Follow the instructions provided with the controller for more detailed information.

8.4 Press Operation

When the chamber has been evacuated and the die reaches the desired temperature:

1. Check to ensure that the advance screw gear box is in the <u>advance</u> position (see section 3.1).
2. Turn the servo motor variac CCW all the way off.
3. Switch the servo control power ON, the large red pilot light should come on.
4. Switch the servo selector switch to the left.
5. Switch the drive direction switch to ADVANCE
6. Slowly turn up the variac until the servo motor can be heard moving, check that the ram is <u>advancing</u> (up). If its retracting instead, turn the variac back to zero and shift the gear box to the advance position.
7. Advance the ram until the motion stops and monitor the load cell output. Increase the variac setting until the required load is obtained.
8. The drive pulley may be manually turned to increase

the load exerted. However, be careful to avoid getting fingers in the way of the belt, as blood will tend to cause belt slippage.

8.5 Shut-Down Procedure:

Following hot pressing:

1. Check the furnace temperature to ensure that it has cooled to an acceptable level, as defined by the pressing program followed.
2. Switch diffusion pump OFF
3. Wait at least 5 minutes
4. CLOSE backing valve, switch backing pump OFF
5. CLOSE diffusion pump coolant valve
6. Switch ion gage OFF
7. OPEN vent valve, allowing chamber to reach atmospheric pressure and open the door.

8.6 Sample Removal

Open the door and loosen the electrical contacts for the front half of the furnace, removing the furnace half subsequently. Lower the ram until the clearance is sufficient and remove the die by rotating and pulling it away from whichever ram it has remained in contact with.

Section 9, Maintenance

9.1 Vacuum Pumps and Fluids

The mechanical roughing and backing pump has an oil capacity of approximately 3.5 liters, and is equipped with an oil level sighting window. The correct level should be indicated at the line in this window with the pump operating. The pump oil used is CVC - 70/19 with a vapor pressure of 8 X 10^{-8} torr. This oil should be filtered or changed every few thousand hours of operation, and the pump cleaned with mechanical pump flushing fluid. Contamination of the pump and fluid, particularly by water vapor, should be carefully avoided.

The diffusion pump has a rated capacity of 1.0 liters, although a note left on the surplus pump indicated that 765 milliliters was the appropriate amount. With a full liter of fluid the time required for pumping to begin is about 5 minutes, greater than the 3 minutes claimed by the manufacturer, and so the 765 milliliter figure may be a better guideline.
This pump is not equipped with an oil level indicating window, and so any oil leaked or vented should be replaced, or the pump removed and the level checked.

The fluid is Convoil 40 which has a very high resistance to oxidation and can tolerate high gas flow rates, but which has a fairly high vapor pressure, 1 X 10^{-5} The flow of coolant through the cooling jacket should not be interrupted, a thermal switch that will shut the pump off in this event may be added.

As with the mechanical pump, contamination of the pump must be avoided. Care should be taken that debris from experiments not enter the vertical pump intake.

9.2 O-Ring Seals

Numerous o-ring seals are employed in the vacuum chamber. Those that are subjected to high service temperatures must be made from Viton, or some equivalent. These particularly include the inner o-rings in the linear motion feedthroughs, which will be particularly hot during long, high temperature pressing cycles. All o-rings must be kept at least lightly lubricated with silicone grease or some equivalent, high vacuum lubricant, and this should be applied prior to installation to reduce the chance on damage. Care should be taken during installation and use to avoid damage such as cuts and abrasions (section 9.3).

9.3 Press Lubrication

The stainless steel rams, and the linear motion feedthroughs should be lightly lubricated with silicon vacuum grease. The screw advance machanism should be kept greased, and the advance gear box should be kept greased. The smaller side-mounted input gear box should be kept filled with a high-grade light machine oil with a low viscosity to minimize

drag.

Section 10. Disassembly and Reassembly

10.1 Press Rams

The upper press ram must be removed first, either by removing the load cell support crosshead, then lubricating the lower end of the uppper ram and sliding it up through the upper linear motion feedthrough, or by removing the entire flange, feedthrough, and ram assembly which is preferable if access to the lower ram and feedthrough is all that is desired.

The rams should always be slid through the feedthroughs slowly, smoothly, and with lubrication, to prevent damage to the o-rings. The lower ram may then be unpinned at the base where it is linked to the screw and slid up and out of the lower feedthrough. The lower feedthrough may be removed may be removed from the lower flange if the screw needs to be removed. The screw may then be driven upward to its limit and the pin at the base that prevents rotation may then be removed and the screw rotated upward manually until clear of the drive gear.

10.2 Electrical Feedthroughs

The electrical feedthroughs involve tightly squeezing elastomer tubing in compression fittings and require the use of Poisson's ratio for assembly and disassembly. The key is to stretch the tubing axially in order to reduce its diameter when clearance on its outside is required, as with the feedthrough fittings, the ferrules, and the compression nuts, and to compress the tubing axially when clearance on its inside is required, as with the brass rod electrical conductors.

The order best followed for assembly, to avoid confusion and because the compression nuts will not clear the feedthrough's chamber wall hole, is as follows:

1. Insert feedthrough through chamber wall with the flange on the inside of the chamber and attach the mounting nut.
2. Lubricate the brass rod or other conductor and slide it into the elastomer tubing, which should be slightly longer than the rod itself . The end of the rod should be smooth and free of sharp edges and the tubing should be <u>compressed</u> to expand its inside diameter and provide clearance. Paper towels will will greatly facilitate this process by providing solid traction on Tygon type tubing, in spite of any lubricants. Leave excess tubing protruding over each end of the rod to proved a grip for the next step.
3. Lubricate the outside of the tubing rod assembly and <u>stretch</u> the tubing to reduce its outside diameter. Pass it through the feedthrough, being careful to keep the tubing extended. The slight excess tubing at the end helps smooth this process.

4. Center the rod and tube in the feedthrough and pass the lubricated compression ferrules over each end. Teflon or other plastic ferrules are recommended over brass. Pass the compression nuts over each end, compressing the tubing in the process to form the seal. A final coat of silicone grease in and around the compression ferrules is a good idea.

5. Tighten the compression nuts firmly, but not too tightly. Tighten them just right.

10.3 Chamber and Column Structure

Disassembly of the chamber and column structure involves first removing the load cell-crosshead assembly, and the upper flange assembly. Chamber removal then proceeds by removing the threaded columns upwardly, leaving the chamber resting on the lower feedthrough mounting flange and the back supports.

The two flange nuts on the upper surface of the interior of the chamber may then be loosened a couple turns, leaving the chamber clamped between the lower flange nuts and the lower feedthrough mounting flange. The nuts beneath this flange then be screwed downward and those beneath the base plate may be removed entirely, releasing the chamber. The top pair of flange nuts should then be screwed downward, till they are near, but not flush to the lower pair, taking care that the o-rings that seal between the flange nuts and the smoothed surface of the threaded columns are left behind at the smoothed surface

The lower flange nuts should then be screwed slightly upward, again leaving the o-rings behind. All four o-rings should then be slid up from the smoothed sections of the thread and over strips of thin manilla cardboard which has been taped tightly over the columns, on the threaded region slightly below each o-ring. These rings will then be slid over the threaded portion of the columns to prevent damage to the rings. By working all the nuts downward and sliding the o-rings along, the columns are then worked upward and free, leaving the chamber resting on the lower feedthrough mounting flange.

Re-assembly of the chamber proceeds by performing the preceeding sequence in reverse order, taking particular care that the o-rings are not damaged.

10.4 Diffusion Pump

The diffusion pump is removed by disconnecting the water and electrical connections and removing the mounting bolts. The bolts are 9/16 inch hollow hex head type and should be held while the nuts are rotated to minimize damage to the o-rings beneath the sealing flanges. Disassembly of the diffusion pump proceeds by first dumping the pump oil, and then unbolting the base and lifting the pump body carefully upward directly vertically to avoid damage to the pump stack.

Section 11. Initial Specimen Production

11.1 Metallic Glass Ribbon - Sn/Pb Matrix

As an initial test of the vacuum hot pressing system's performance, two metallic glass ribbon reinforced, metal matrix composites were fabricated. The metallic glass employed was an iron/silicon alloy while the matrix was a eutectic composition of tin and lead (63/37 Sn/Pb). Chill cast ribbon was cut into strips 1.0 cm X 1.5 cm and laid up alternately with rolled strips of the Tin/Lead alloy. Both the metallic glass ribbons and the matrix strips were lightly scraped with 600 grit silicon carbide paper, but no degreasing was performed and recontamination of the surfaces due to fingerprints and other sources was evident.

The specimen was pressed at a temperature of approximately 275 xC, with a vacuum of 2 X 10^{-4} torr and under a load which was not measured because the load cell was not yet connected, but which was in the range of 500 Kg for a pressure in the ram of approximately 10 mPa.

Melting of the matrix material occurred, leaving the metallic glass ribbons tightly compacted together in a high volume fraction composite (approx. 95%), (figure 11). The interface strength was very, as would be expected with these minimal surface preparations, and delamination easily occurred. No attempt was made to determine whether the glassy nature of the ribbons was maintained due to the were probably sufficiently high to cause crystallization in this alloy.

11.2 Metallic Glass Ribbon - Bi/Sn/Pb Matrix Composite

Strips of the same metallic glass were cut donw to .5 cm X 1.5 cm and laid up in a staggered, laminated pattern with rolled out sheet of a bismuth, tin, lead eutectic alloy with a melting point of 124 xC. Surface preparations were the same as those outlined above.

Pressing proceeded with a peak temperature of approximately 75 xC, a press load in the range of 500 Kg (10 Mpa), and under a vacuum of 2 X 10^{-4} torr for 30 minutes.

Extensive flow occurred and a laminate was formed (figure 12), although interface strength was again very low.

Again, no x-ray or DSC analysis was performed to check for retention of the glassy nature of the ribbons, however, at this low temperature regime crystallization probably did not occur.

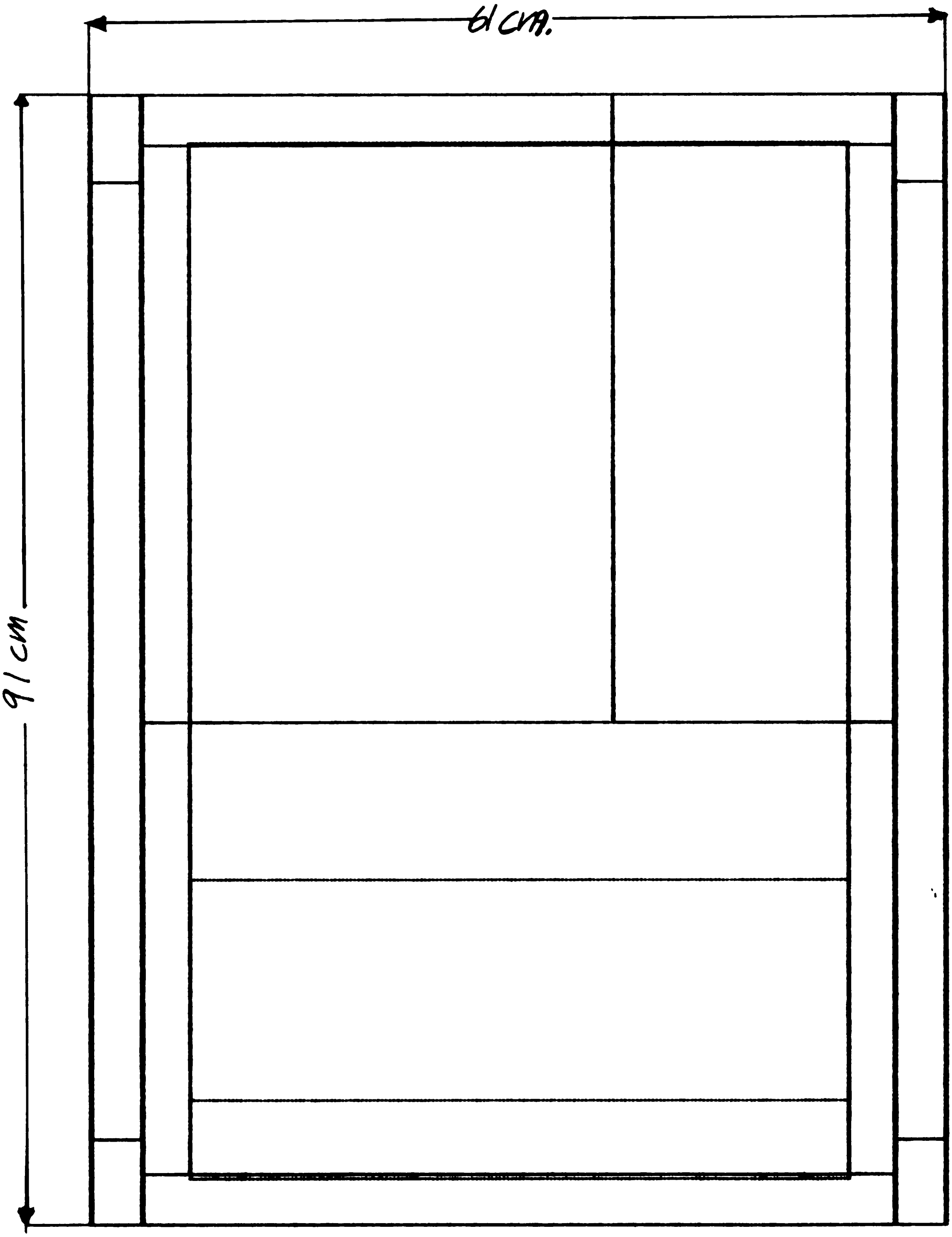


Figure 1. Top View of Chassis and Frame

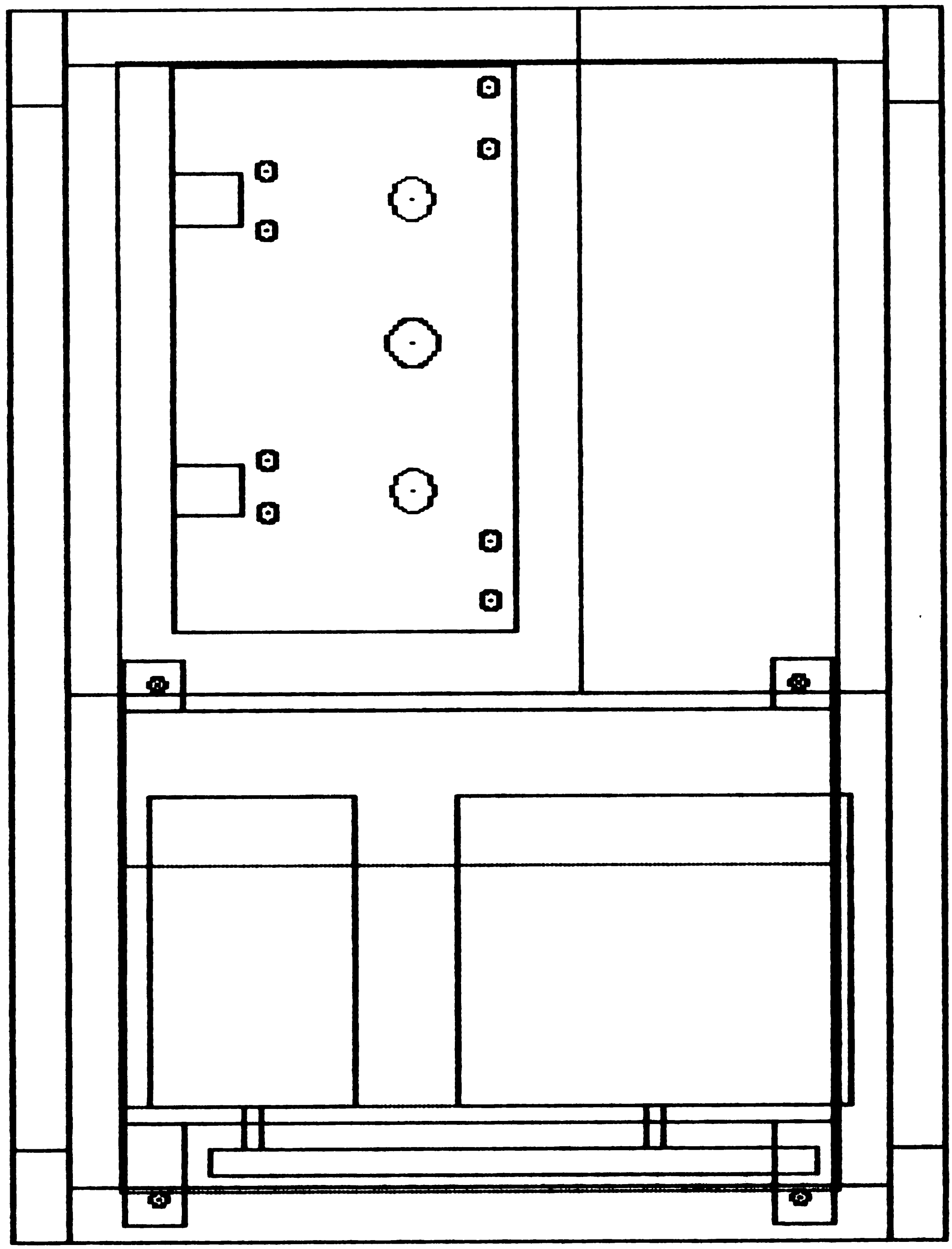


Figure 2. Top View With Base Plate and Mechanical Pump

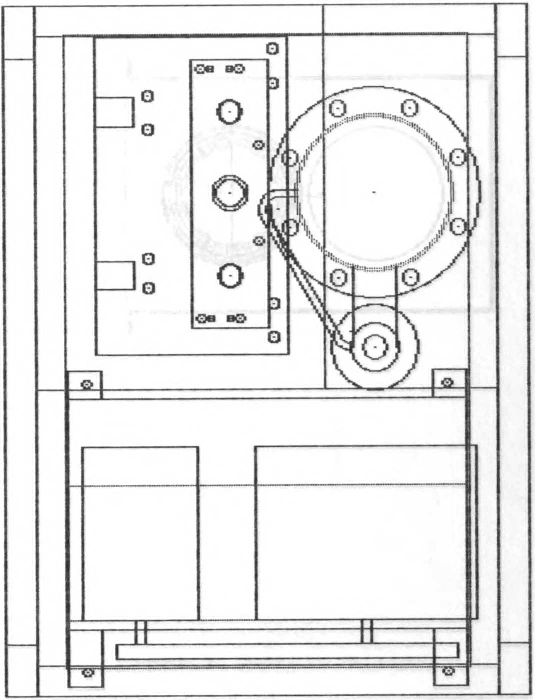


Figure 3. Top View With Flange and Diffusion Pump

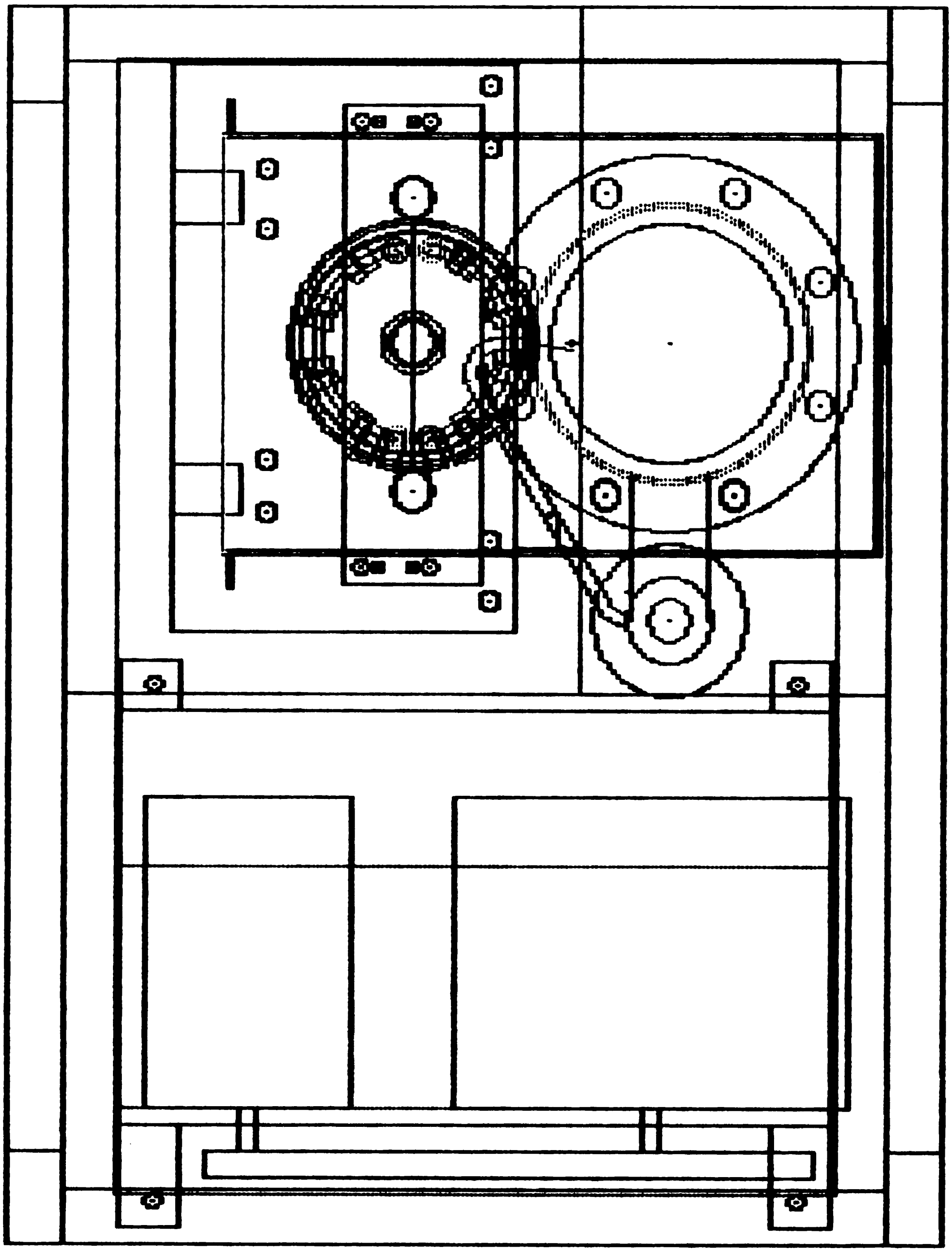


Figure 4. Top View With Chamber and Furnace

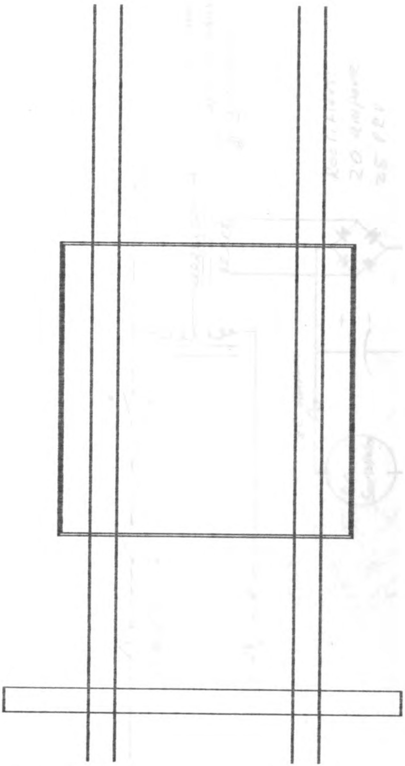


Figure 6. Front View of Vacuum Hot Press

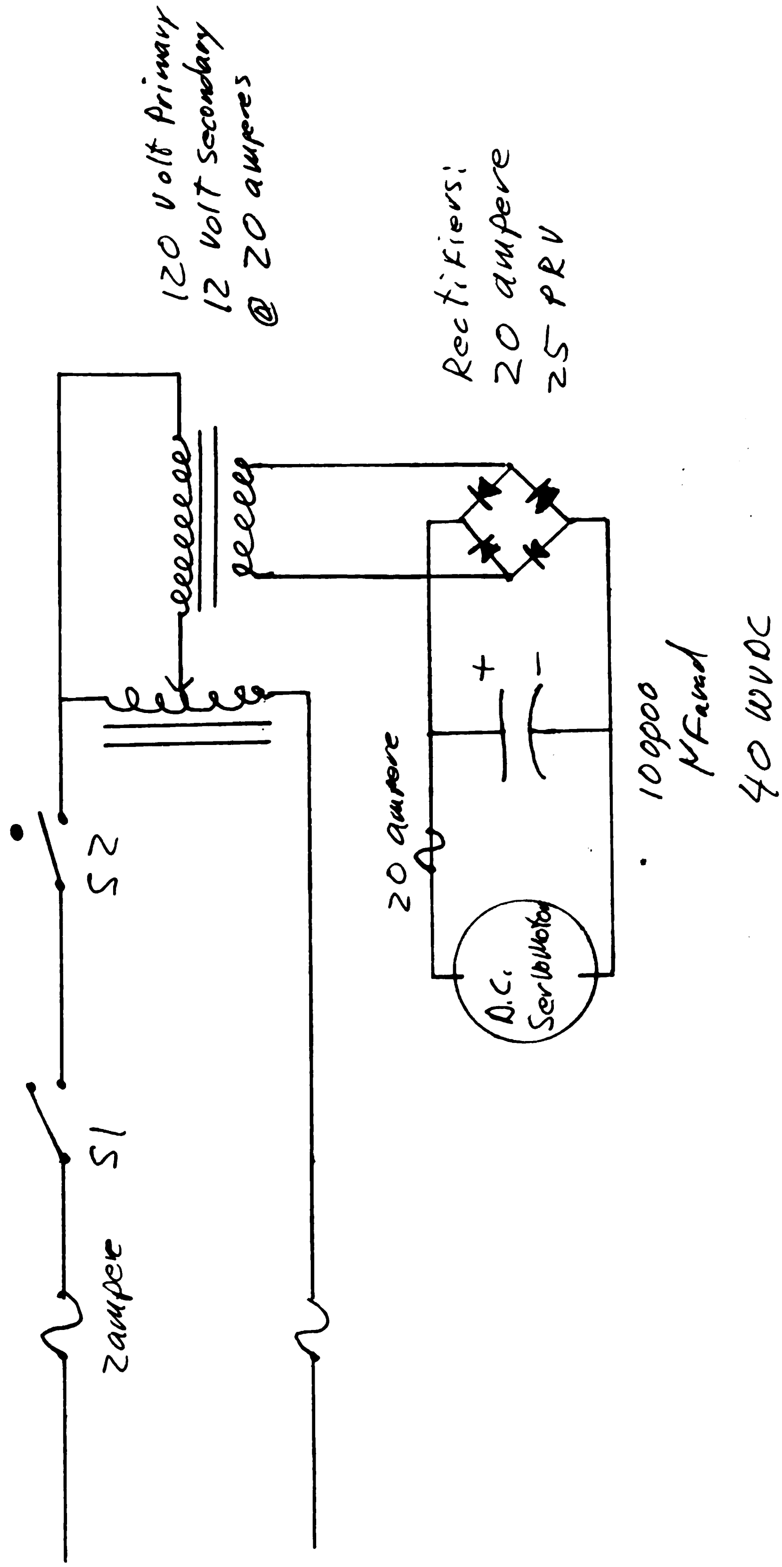


Figure 6. D.C. Servo Motor Circuit

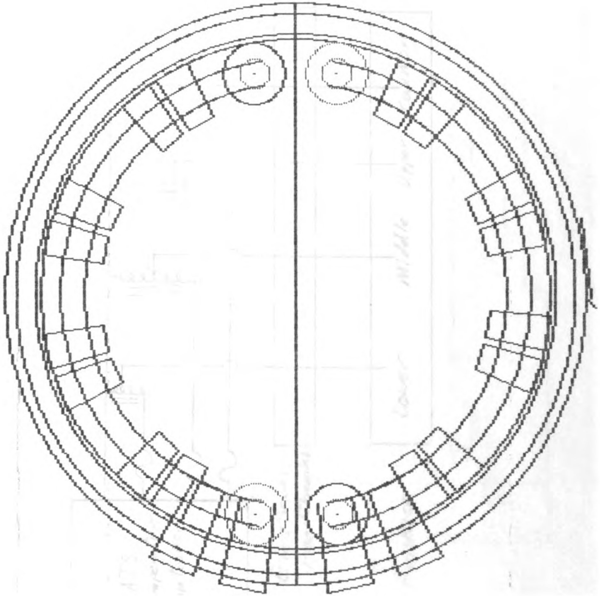


Figure 7. Top View of Furnace Heating Elements

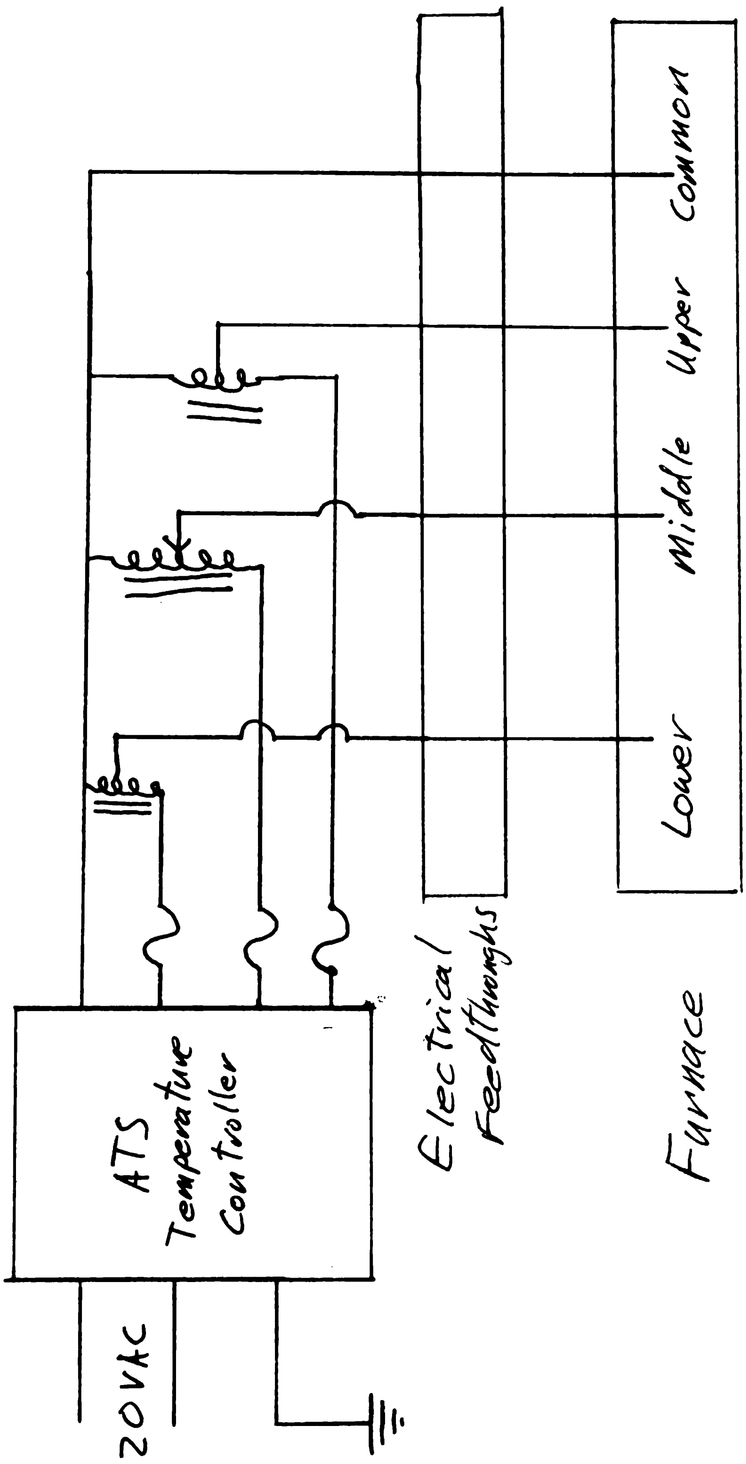


Figure 8. Furnace Control Circuit

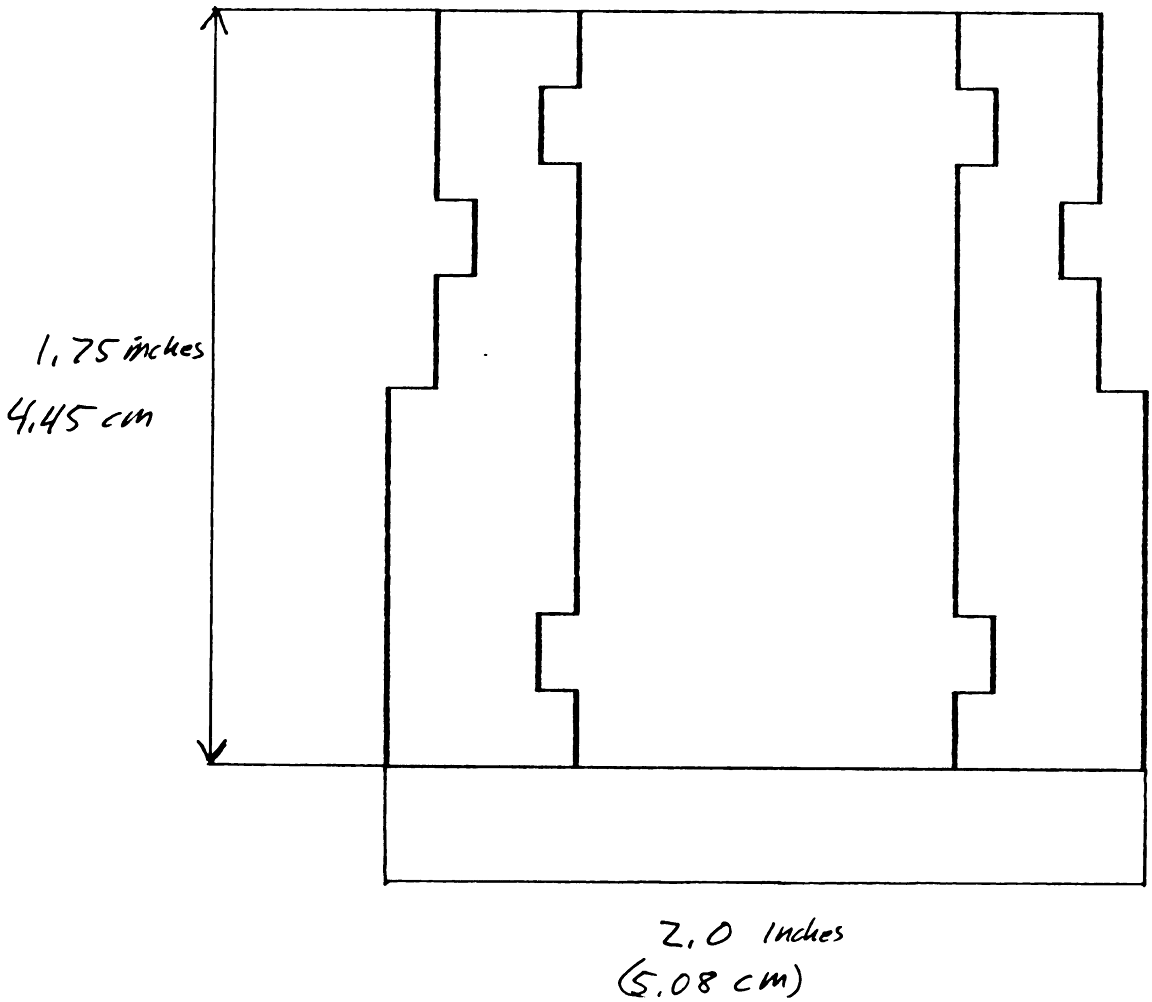
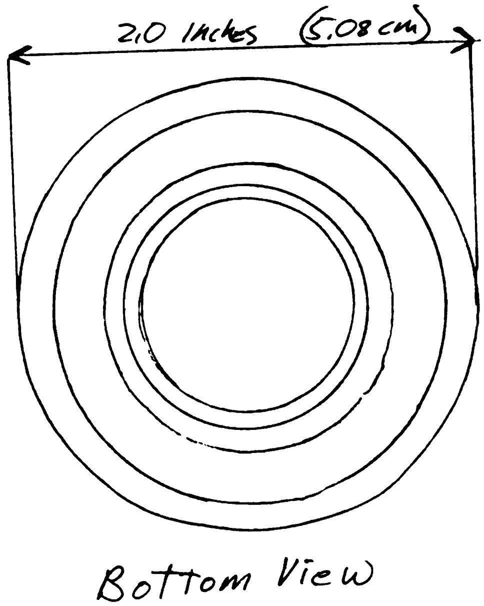


Figure 9. Linear Motion Feedthroughs



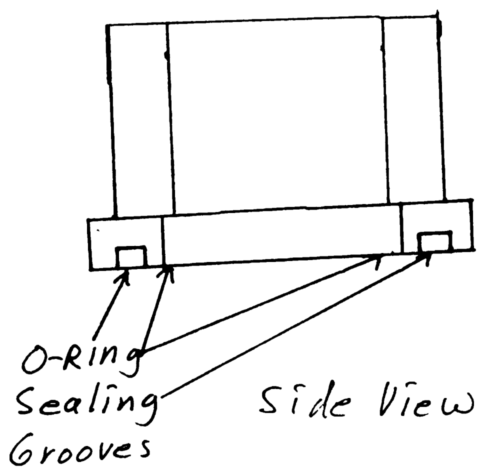


Figure 10. Threaded Column Feedthroughs

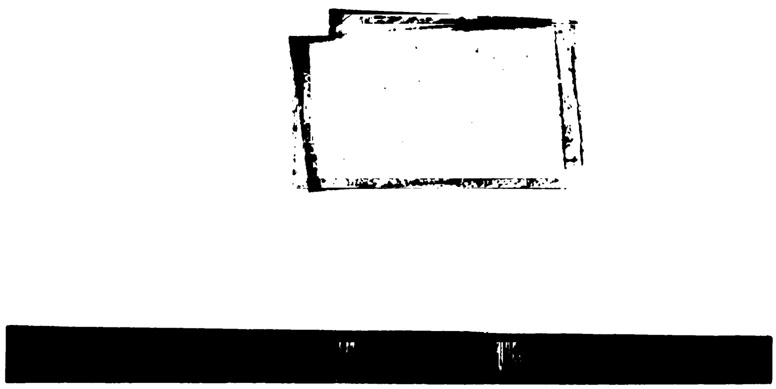


Figure 11 Metallic Glass Ribbon - Sn/Pb Matrix Composite

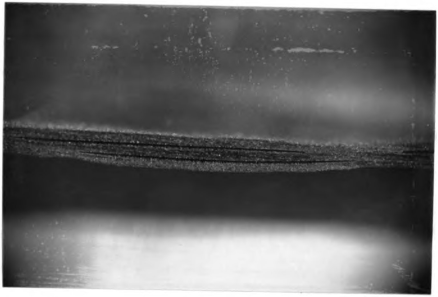


Figure 12 Metallic Glass Ribbon - Bi/Sn/Pb Matrix Composite

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