Design and Construction of High Temperature Uniform Metal Spray Apparatus

by

Godard Karl Abel

Submitted to the Department of Mechanical Engineering
in Partial Fulfillment of the Requirements
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Signature of Author ______________________________
Department of Mechanical Engineering
December 21, 1992

Certified by ______________________________
Professor Jung-Hoon Chun
Thesis Supervisor

Accepted by ______________________________
Professor Peter Griffith
Chairman, Undergraduate Thesis Committee

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Abstract

An apparatus was designed and built to create uniform metal droplet sprays for temperatures up to 1200° C. The design of the apparatus was based on an existing uniform metal spray forming apparatus that was developed by Passow [1992]. The original apparatus could spray metals with melting points below 450° C and showed that it was possible to create sprays of uniform metal droplets. The new apparatus was developed to expand the range of metals that can be sprayed and to improve the robustness of the original design.

The goals in the design process were to make the new system robust, easy to assemble and control, while minimizing the cost and fabrication time for the apparatus. Several concepts were considered for the design of the furnace, pressure, and vibration systems. The electric resistance furnace and single pressure chamber concept was chosen based on its ability to meet the design goals. The system uses the same monitoring and control system as the original apparatus, but all the other hardware had to be redesigned and constructed. The metal is melted inside a high-density graphite crucible by clamshell electric resistance heaters. The crucible is sealed by flexible graphite gaskets and can be pressurized up to 70 KPa to spray a molten metal jet through 50μm and 100μm orifices. A vibration is imposed on the jet using a piezo-electric vibration mechanism and this breaks the jet into uniform droplets with diameters between approximately 70μm and 220μm. As the jet breaks up into droplets, it passes through a graphite charge ring, which has a high positive electric potential between 250 and 400 Volts. The droplets retain an equal negative charge, which keeps the droplets separate in flight. The droplets are sprayed into a one meter long boro-silicate glass chamber and can be collected on a substrate anywhere in this chamber. The spray chamber and furnace are sealed from the atmosphere with PTFE and Viton O-rings and can be pumped down to a 10^{-4} torr using a mechanical vacuum pump. The system is filled with nitrogen during tests to prevent the oxidization of the molten jet.

Some initial melting, and spraying tests were performed using a 90% Cu and 10% Sn powder, and these tests showed an ability to melt and spray copper alloys. After initial testing a design modification had to be made to reduce the stress on a ceramic ring that supports the crucible. The system will be used to produce uniform metal powders and to study the basic principles of uniform metal sprays. Eventually the apparatus will be used to study uniform coating, spray welding, near-net shape forming, and metal matrix composite production.

Thesis Advisor: Professor Jung-Hoon Chun

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1. Introduction

An apparatus has been designed and fabricated that will enable the Droplet-Based Manufacturing (DBM) group in MIT's Laboratory for Manufacturing and Productivity to investigate the uniform metal droplet spraying of high melting temperature metals. We have an existing apparatus designed and built by Passow [1992] that creates uniform metal droplet sprays of lower melting point metals such as tin, zinc, lead and their alloys. The DBM process uses the same concept to create uniform droplets that is used in continuous ink jet printing. A molten metal jet is broken up by imposing forced vibrations on the stream, and therefore it breaks up into droplets with diameters uniform within 3%-4%. The droplets are charged electrically to the same negative charge, and therefore remain separate in flight.

The goal of this study was to expand the capabilities of uniform metal droplet sprays by designing a new system that would allow us to melt metals with melting points up to 1200° C. This will allow us to spray metals such as aluminum, copper and their alloys. The other major goals of the design process were to make the system as robust and easy to assemble as possible, while minimizing the cost of the apparatus. The major engineering challenge was to design the system so that it could withstand the combination of high temperatures and high pressure.

Uniform metal sprays overcome problems in traditional metal spray forming processes, in which gas atomization is used to break up the droplets. In conventional spray forming the droplet diameter varies significantly. As a result of the uniformity of the droplets, the DBM process allows precise control of mass and enthalpy flux and ensures that all droplets have similar temperature histories and uniform velocity profiles. These properties make it possible to control the microstructure of spray deposits. The potential applications for uniform metal droplet sprays include the following: uniform metal powder production, uniform spray coating, near-net shape forming of billets, metal-matrix composite production, and fabrication of complete three-dimensional parts.

In the next section of this thesis the process and existing low-temperature apparatus are described in more detail. In Section 3, the conceptual design of the high temperature apparatus is reviewed. This is followed by a complete description of the components of the high-temperature system. In Section 5, the results of initial testing are discussed and design modifications are outlined. The future applications of the high temperature apparatus are described in the conclusion.

2. DBM Process and Original Apparatus Description

The DBM process uses the same mechanism to spray uniform droplets that is used in continuous ink jet printing process, but it incorporates major modifications to spray molten metals. The first step in the DBM process is to melt the metal by applying heat to a crucible containing the metal, as can be seem in Figure 2-1 [Passow, 1992]. Then pressure has to be applied to the crucible to force molten metal through an orifice at the bottom of the crucible and thereby form a jet of molten metal . The jet is then broken up into uniform droplets by vibrating a disk which is immersed in the molten metal near the orifice. The vibrations are created by a piezo-electric transducer crystal ,which vibrates at a set frequency, and the vibrations are transmitted from the piezo-electric transducer to the disk by a shaft. This creates a sufficient disturbance to break up the jet at the desired frequency. This frequency can be used to control droplet diameter. At the point where the jet breaks up into droplets, a negative electric charge is applied to each droplet so that the droplets will repel each other during flight. This prevents the droplets from merging further down in the stream.

A schematic diagram of the original melt-vibration system is shown in Figure 2-1. The system was designed to melt metals with melting points up to 450 °C. The crucible is made of stainless steel and the heating elements are two 300 Watt electric resistance band heaters (Omega MB-300W) and are controlled by an Omega 9000A PID temperature controller. The piezo-electric transducer is separated from the melt, since the transducer can only function up to half of its Curie temperature, which is approximately 200°C. If the crystal is heated to above this temperature, then it loses its piezoelectric behavior. The shaft and disk of the vibration transmitter are made of stainless steel and are immersed in the molten metal to transfer the vibration.

The electric charge is applied to the droplets using inductive charging. A charge plate, which is made out of brass, is attached to the bottom of the crucible. It has a small slot through which the metal jet passes. The charge plate is positioned so that the jet breaks up into droplets, in the region where it passes through the charge plate. The charge plate has a high positive voltage, while the crucible is grounded. The individual droplets are inductively charged by this difference in electric potential and the capacitance between the droplets and the charge plate. All the droplets receive the same negative charge, and therefore repel each other in flight. The charging plate has a DC voltage of between +250 and +400 Volts, and through inductive charging creates a droplet charge between .05 and .1 Coulombs/gram for 200μm droplets.

The entire original apparatus is shown schematically in Figure 2-2. The crucible is enclosed in a Pyrex chamber which is filled with nitrogen. The nitrogen provides an inert atmosphere for the spray, which is required to prevent oxidization of the molten jet as it leaves the orifice. If an oxide layer forms on the jet, this prevents the breakup of the stream into droplets. The crucible is pressurized with nitrogen and this forces the metal through the orifices. The metal sprays are then collected on a substrate which can be positioned anywhere in the spray chamber.

The control and monitoring system are also shown in Figure 2-2. The piezo-electric vibration is controlled by a frequency generator which is monitored by an oscilloscope. The output from the function generator is amplified and the voltage is stepped down through a transformer before it connected to the piezo-crystal. The temperature is controlled by an Omega 9000A PID Controller and the voltage on the charging plate is set on a Bertran High DC Voltage power supply (Model #230). The chamber and crucible pressure are controlled using two nitrogen tanks and a pressure regulation system. A vacuum pump is used to flush the system and minimize the oxygen content in the chamber. To record the droplets a video camera and a strobe light are used. The strobe light is set at a frequency equal to the droplet breakup frequency so that the individual droplets are captured on the videotape.

The original apparatus has produced uniform droplets using tin, tin-lead alloys, and zinc. It also has produced uniform metal droplets with up to twelve orifices. The original apparatus showed that the DBM process could be used with different metals and with multiple orifices, but it is limited to metals with melting points below 450° C. There are also problems with the original system in terms of robustness and ease of use. The sealing of the original system is often a problem and these leaks cause oxidization of the jet. For spraying tin, which has a melting point of 229° C, the low-temperature system works consistently. For spraying zinc, which has a melting point of 419° C, the system performs less consistently. The higher temperatures lead to O-ring sealing problems, and oxidization of the jet occurs more frequently. This makes it difficult to produce uniform droplets consistently. The zinc is also very difficult to remove from the crucible and vibration shaft after experiments, since it adheres to the stainless steel.

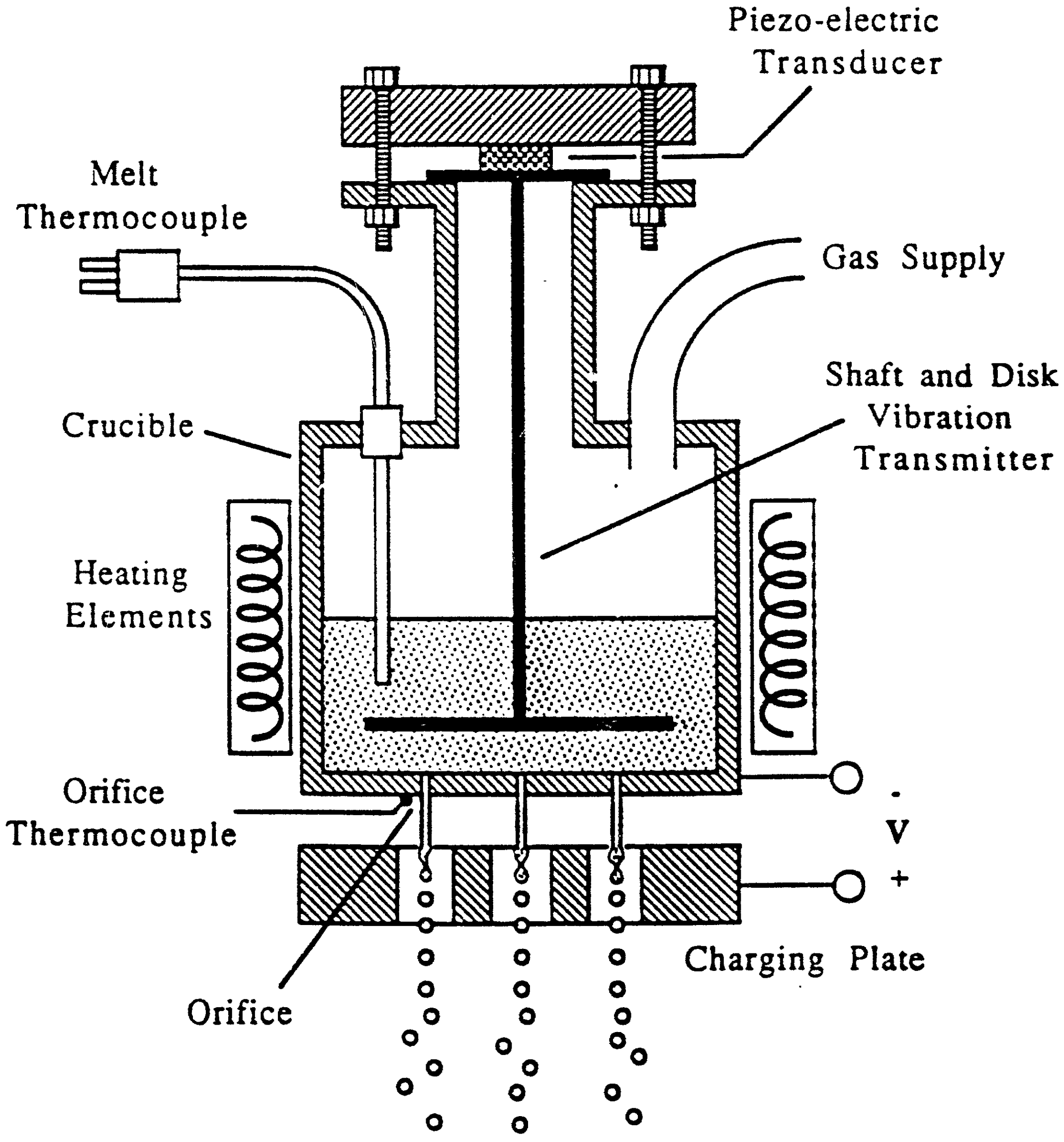

Figure 2-1: DBM Process Schematic [Passow, 1992]

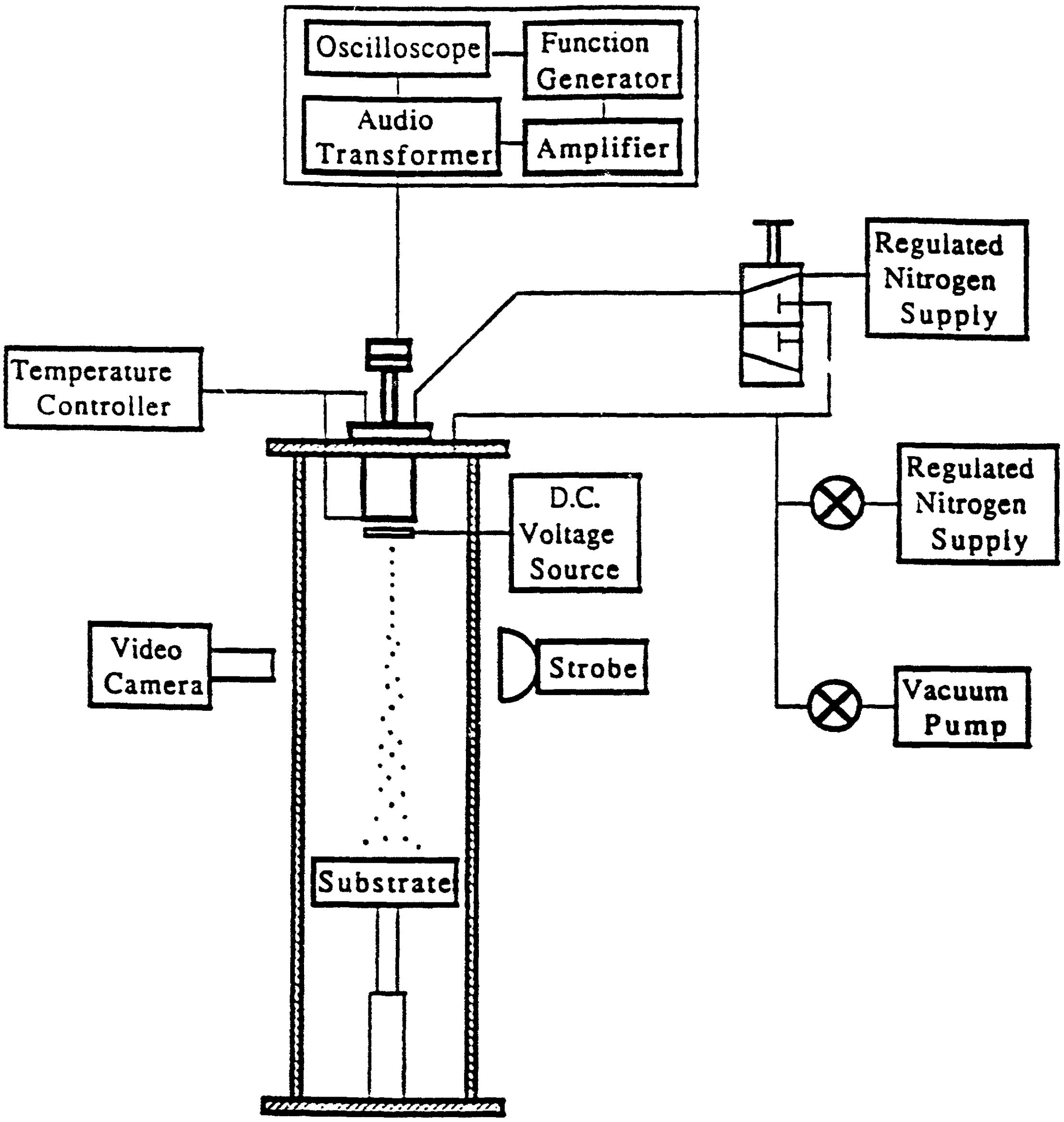

Figure 2-2: Schematic Diagram of Low-Temperature Apparatus [Passow, 1992]

3. Conceptual Design of High-Temperature System

In order to melt and spray metals with melting temperatures up to 1200° C, the entire apparatus has to be redesigned. The goals for the system design were to make the system robust and easy to assemble and control. The other objectives were to minimize the cost and fabrication time of the system. Two key issues had to be decided. One was the method of heating and the other was the method of containing the pressure that needed to be applied to the melt. Finding a way to achieve the necessary temperature and simultaneously containing the pressure was the primary challenge in the design.

Several different concepts were considered for the new system. These concepts included: an induction heating furnace with two separate pressure chambers, an arc heating furnace, and an electric resistance heating furnace. The concepts were evaluated on the basis of how well they could satisfy the design goals.

One concept was to use induction heating coils with a ceramic crucible as shown in Figure 3-1. The entire crucible and coils are enclosed in a water cooled stainless steel chamber. The entire chamber is pressurized to force the metal through the orifices into the spray chamber, which is kept at a lower pressure. The seal between the spray chamber and stainless steel chamber is made using a flexible graphite gasket at the bottom of the crucible. This design minimizes the strength requirements on the crucible, since it only has to support the pressure over the area of the hole in the stainless steel section.

The concept of an arc furnace with a ceramic crucible was also briefly considered; however, the precise control needed to keep the arc between an electrode and the melt would have been very difficult and expensive. Therefore, the concept was not seriously considered. For a system that would go to temperatures high enough to melt iron and steel (approximately 1800°C) an arc furnace might be the best solution, since clamshell electric resistance heaters cannot reach the required temperature.

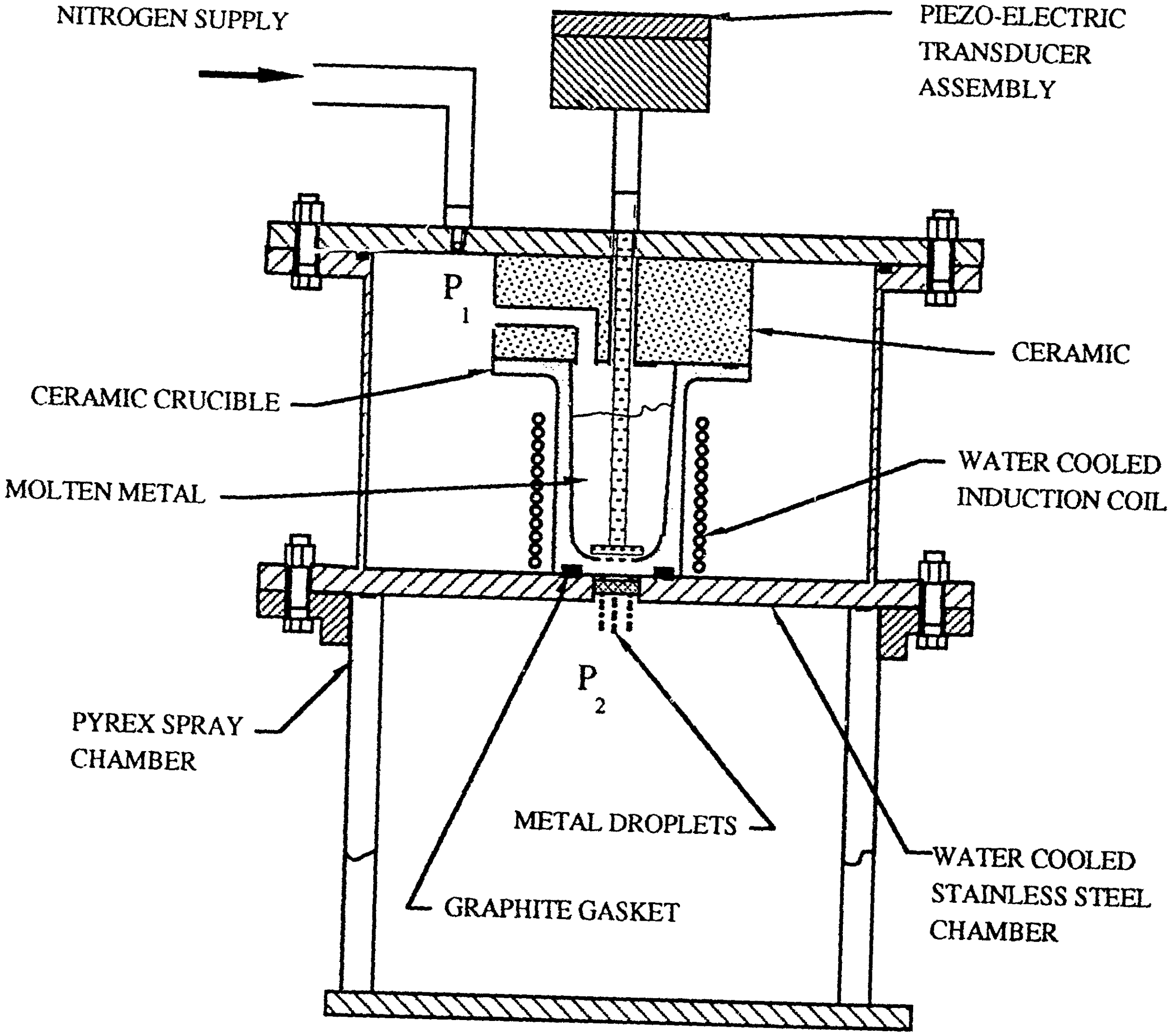

Figure 3-1: Induction Furnace and Two-Chamber Concept

The third concept was to use electric resistance clamshell heaters that would heat the crucible by the means of radiation and convection. In this concept, which is shown in Figure 3-2, the crucible is made out of graphite, which has a high thermal conductivity. The entire furnace section is insulated with ceramic fiber insulation which keeps the heat inside the crucible. A ceramic ring is used to support the crucible and insulate the bolts from the heat source. In this concept the crucible is pressurized and has to have sufficient tensile strength to withstand this pressure. The seal between the spray chamber and crucible is made using a flexible graphite gasket that has the a maximum operating temperature of over 2500 °C.

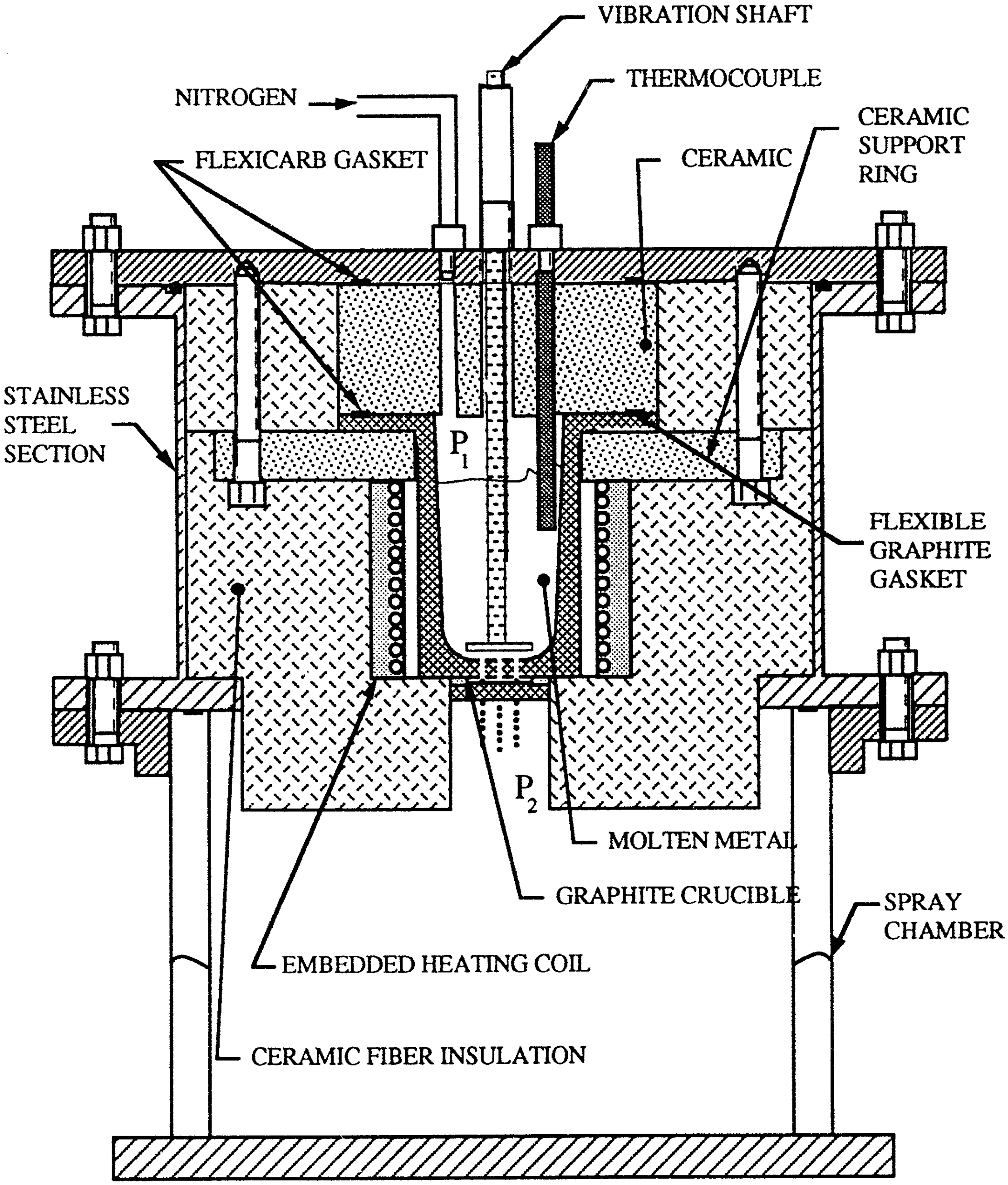

Figure 3-2: Electric Resistance Furnace Concept

Both the induction heating concept and electric resistance concept were seriously considered and evaluated with respect to the design goals. The robustness of the two systems was difficult to predict, but appeared to be similar for the two designs. The key issue in a robust design is to minimize the oxygen level in the chamber and this could be achieved equally well with the two designs. In terms of ease of assembly the electric resistance system had a slight disadvantage, since it requires the ceramic ring and crucible to be bolted to the top plate. In order to refill the crucible the entire system has to be disassembled.

The cost of the two systems was the major deciding factor. A water-cooled stainless steel chamber would have been more expensive than a standard stainless steel section. The induction heating process is also very inefficient since much of the heat is lost to radiation to the water cooling the induction coils and chamber. The induction heating supply we had in our laboratory is a high frequency unit, which therefore has little skin depth, and it would not have heated the metal directly but would have heated the crucible first. A non-conductive ceramic crucible would not have transferred enough heat to the metal. This means that a conductive material such as graphite would have been required, which would have lead to greater heat losses to the chamber. For the melting of metals a medium to low frequency induction unit is preferred, since a stirring effect is created that helps to melt the metal [Davies, 1979]. Purchasing a new induction power supply would have cost between $10,000 and $20,000. The electric resistance clamshell heaters could be purchased for less than $200. In terms of fabrication time the electric resistance system allowed the use of more standard components, and therefore the fabrication time was reduced. On the basis of this analysis the electric-resistance heating system was chosen and the final design is described in detail in the next section.

4. Detail Design of High-Temperature System

The new apparatus is shown schematically in Figure 4-1. The monitoring and control systems have been kept nearly the same, but all of the other components have been redesigned. To provide enough heat to melt high-temperature metals a furnace section was added to the top of the spray chamber. All the components had to be designed to withstand the high temperatures and provide a tight seal to the atmosphere. The major components of the system include the crucible, furnace and temperature control system, piezo-electric vibration system, spray chamber, and sealing system. The design of each of these major components is detailed in this section.

4.1 Crucible

The crucible was critical in selecting a design that could take the high temperatures and seal against pressure. The material selection and strength calculations for the crucible are outlined in this section.

4.1.1 Material Selection

The material for the crucible was selected first. A high density graphite (Toyo Tanso, Grade: IG-11) was chosen, since graphite has a very high thermal diffusivity due to its high thermal conductivity and low thermal capacity. This means that the heat is quickly transferred from the clamshell heaters to the metal in the crucible. The IG-11 has a thermal diffusivity (thermal conductivity/specific heat) value of .3 m^2/hr [Toyo Tanso Co., 1992]. The other feature of graphite is that it is a material that gets stronger as temperature is increased. In an inert atmosphere graphite can be heated to 2500° C and have a strength higher than its strength at room temperature. At 1200 ° C, which is the maximum operating point, graphite is approximately 1.2 times as strong as it is at room temperature. Another key factor in selecting the material was that it is not wetted by the molten metal. This means that the molten metal does not stick to it, and the metal can be easily removed from the crucible after experiments. The only drawback of graphite is that it will oxidize above 350 °C, if it is not kept in an inert atmosphere. However, the DBM process requires an inert atmosphere for the metal spraying, so it is not an added requirement.

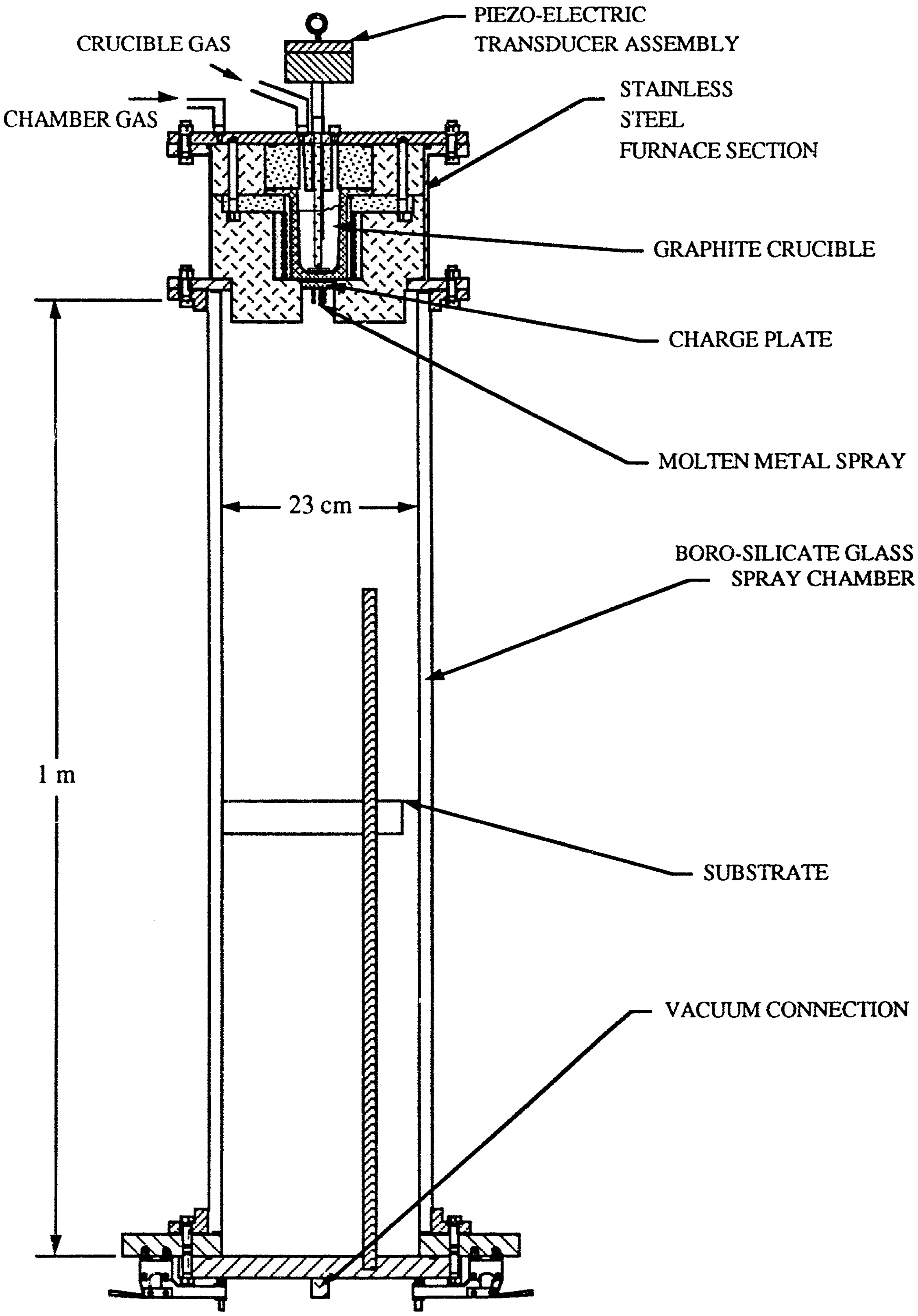

Figure 4-1: High-Temperature System

4.1.2 Crucible Dimensioning and Strength Calculations

Once the material was selected the dimensions of the crucible had to be determined. The crucible needed to have enough internal volume to hold enough metal to run complete experiments. A minimum volume of 100 cubic centimeters was selected, since the existing crucible had this volume, and it allowed runs of up to ten minutes with a single orifice. By choosing inside dimensions of five centimeters radially and ten centimeters long, as shown in the detail drawing of the crucible in Figure 4-2, a volume of approximately 125 cubic centimeters was achieved.

The crucible has to be strong enough to withstand the pressure required to spray the metal. The crucible was designed for a maximum pressure of 700 KPa, which is well above the usual operating point of approximately 200 KPa. To calculate the required thickness of the crucible walls, the crucible was modeled as a thick-walled cylinder. A thick walled cylinder is defined as a cylinder whose wall thickness, t_w, is greater than 1/10 of the crucible diameter, d_c:

$$t_w > \frac{d_c}{10} \tag{4.1}$$

The maximum circumferential stress occurs at the inner surface, σ_Φ, and is equal to the following [Avallone,1987]:

$$\sigma_\Phi = \frac{PR_i^2}{R_o^2 - R_i^2}\left(1 + \frac{R_o^2}{R_i^2}\right) \tag{4.2}$$

where P is the internal pressure, R_i, is in the inner radius crucible , and R_o, is the outside radius. This stress acts in tension. The radial stress at the inner surface is

$$\sigma_r = \frac{PR_i^2}{R_o^2 - R_i^2}\left(1 - \frac{R_o^2}{R_i^2}\right) \tag{4.3}$$

and acts in compression [Avallone,1987].

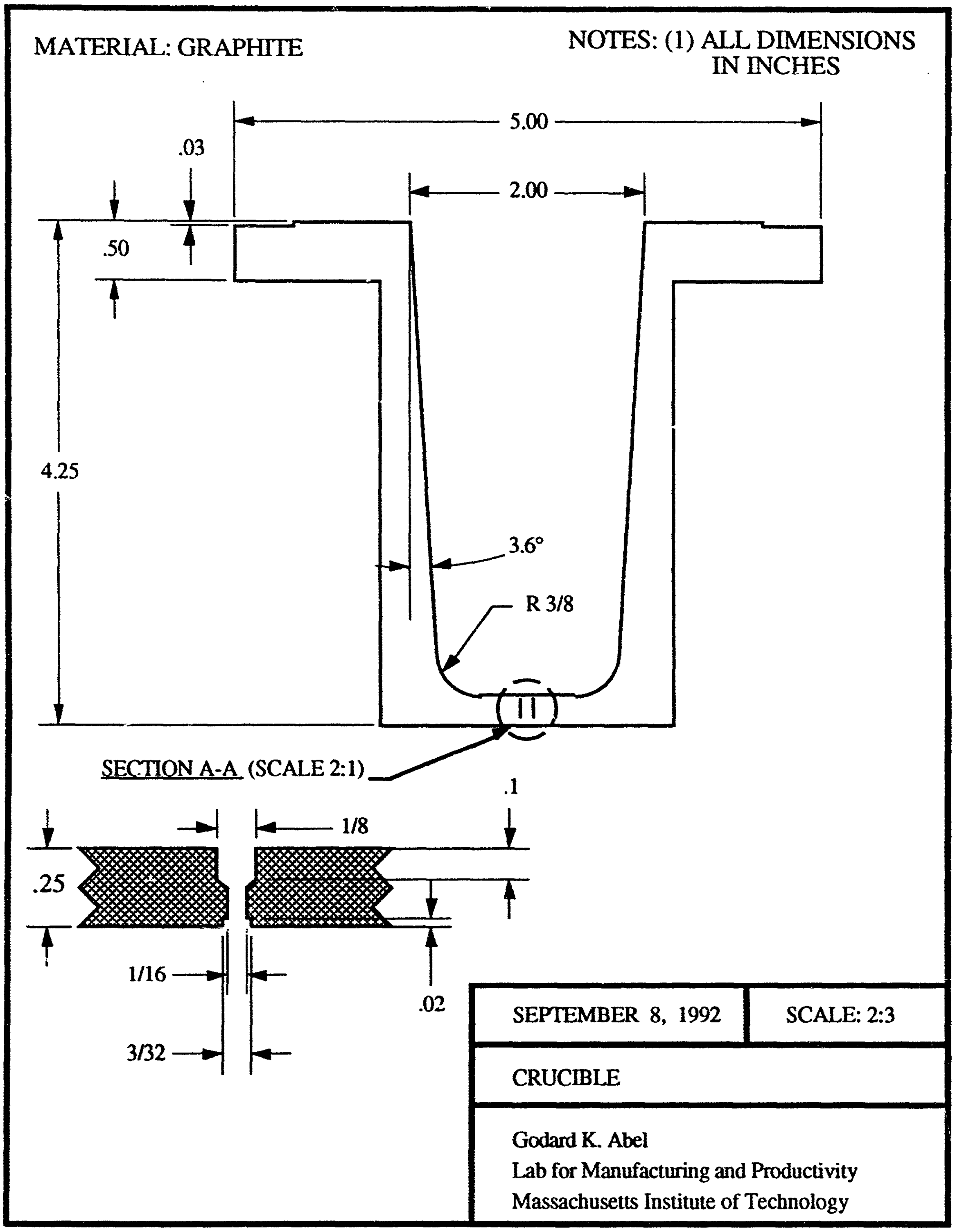

Figure 4-2: Detail Drawing of Crucible

The maximum shear stress is found using from these two principal stress and is equal to:

$$\tau_{max} = \frac{\sigma_1 - \sigma_2}{2} = \frac{P R_o^2}{R_o^2 - R_i^2} \tag{4.4}$$

By making the cylinder walls at least 6.5 millimeters thick the maximum calculated tensile stress was 3.1 MPa, which is well below the tensile yield strength of 27 MPa.

The maximum stresses in the crucible occur in the bottom surface of the crucible. For a flat head at the end of pressure vessel the ASME code [Megyesy, 1975] prescribes a minimum thickness equal to:

$$t = d_c \sqrt{\frac{.2\,P}{S\,E}} \tag{4.5}$$

where S is the tensile strength of the material and E is the joint efficiency between the cylinder and head, which is assumed to be one since the crucible is cut out of one piece of graphite. By substituting these values one obtains a minimum thickness of 3.5 mm for a pressure of 700 KPa. The code also requires a minimum radius of at the bottom of the crucible of 1 cm, which is included in the design.

The other factor that has to be considered is the stress concentration at the hole in the center of the crucible where the orifice is mounted. The maximum stress in the crucible head is given by the following formula from if fixed end conditions are assumed [Avallone, 1987]:

$$\sigma_{max} = (.75)\,\frac{P R_i^2}{t^2} \tag{4.6}$$

The stress concentration factor for a single hole in the center of a round flat plate is a factor of two. However, the crucible was designed with multiple orifices in mind. The crucible was designed for the pattern of orifices shown in Figure 4-3. According to Peterson [1974], this pattern leads to a stress concentration factor of approximately 2.4. Therefore the maximum tensile stress in the crucible head would be:

$$\sigma_{max} = 2.4\,\sigma = 1.8\,\frac{P R_i^2}{t^2} \tag{4.7}$$

By substituting a pressure value of 700 KPa and the dimensions of the crucible shown in Figure 4-3 the maximum stress is 19.3 MPa, which leaves a safety factor of greater than 1.6 in the design.

A taper was also added to the crucible to make the removal of the metal easier and to reduce the stress on the bottom of the crucible. The final dimensioned design of the crucible is shown in Figure 4-3. The small lip on the flange is used to locate the flexible graphite gasket.

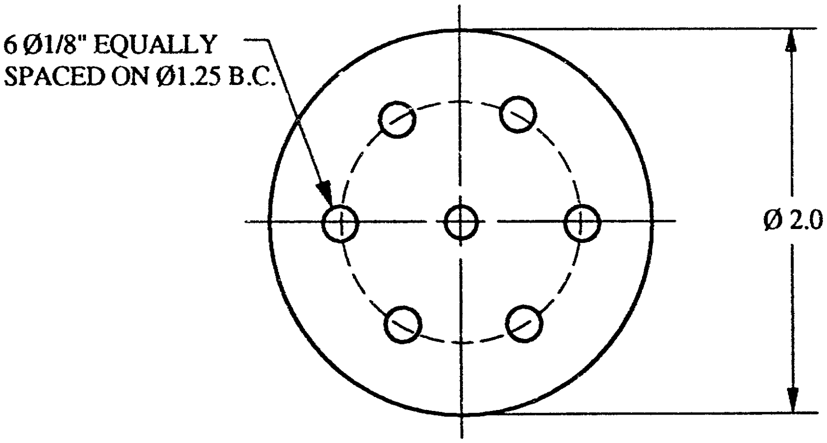

Figure 4-3: Orifice Pattern

4.1.3 Orifice Mounting

The molten metal is sprayed through orifices with nominal diameters of 50 µm and 100 µm (actual diameters of 55 µm and 88 µm, respectively). The orifices are cut into ruby crystals which are grown from an aluminum-oxide base. In order to mount the orifice on the crucible the crystal has to be attached to the crucible. In Figure 4-2, the pocket used to mount the orifice is shown in Section A-A. The small crystal is placed into the small 2.4 mm pocket at the bottom of the crucible and glued with a graphite based adhesive made by Aremco (Graphi-Bond 669). The adhesive holds the orifice on the crucible and prevents the metal from leaking around the edges of the orifice.

4.2 Droplet Solidification Rate

To determine the minimum length of the spray chamber to allow droplet solidification, a simple heat transfer model derived by Passow [1992] was used. The droplets are modeled as individual spheres with an air flow around them. Heat is lost from the droplets in flight by forced convection and also by radiation. This can be modeled by the following equation where the first term represents the convection and the second term the radiation loss.

$$Q = h A_d (T_d - T_g) + \sigma \varepsilon A_d (T_d^4 - T_g^4) \tag{4.8}$$

where h is the heat transfer coefficient, A_d is the surface area of the droplets. The σ represents the Stefan Boltzman constant and ε is the emissivity of the metal. The heat transfer coefficient is taken for the empirical correlation for forced convection around a sphere.

$$h = \frac{k_g}{d_d} (2.0 + Re^{1/2} Pr^{1/3}) \left(\frac{c_{g(avg.)}}{c_g} \right)^{.26} \tag{4.9}$$

K_g is the thermal conductivity of the gas d_d is the droplet diameter, Re represents the Reynold's Number and Pr is the Prandtl number. C_g is the heat capacity of the gas at room temperature and $c_{g(avg.)}$ is the heat capacity of the gas at the average temperature of the droplet and the gas.

Passow [1992] created a numerical model that estimated the cooling rate for tin metal. He neglected the radiation term, since it was insignificant for low-melting point metals such as tin. For high melting point metals such as copper the radiation term is more significant. For a 175 μm copper droplet with an assumed initial velocity of seven meters per second the initial forced convection term is approximately six times greater than the radiation term. This is assuming ideal black body radiation from the droplet to the glass spray chamber, which is assumed to be at room temperature. Passow's model also corrects for the loss in convective heat transfer due to the stream of droplets. The droplets partially shield each other from the drag forces, and therefore lose less heat to the atmosphere than an individual sphere.

Since the radiation loss is still much less than the convective loss Passow's model should give a reasonable approximation for copper sprays. The model will show a solidification distance greater than the actual distance. Copper has a solidification rate similar to tin since its heat of fusion is approximately four times the heat of fusion of tin. Copper also loses heat about four times more rapidly due to the much larger temperature difference between the droplet and the gas. Since 178μm tin droplets solidify completely in approximately eighty centimeters, copper droplets solidify in a slightly shorter flight distance. With a spray chamber length of 100 cm the droplets should be completely solidified before they reach the bottom of the chamber.

Aluminum has a much higher heat of fusion than copper and a lower melting point. Therefore aluminum droplets will solidify much more slowly than copper droplets and will have to be collected in a water bath or another cooled substrate, if powder collection is desired. Otherwise the droplets will still be molten and splatter on impact, which would be desired in some other applications.

4.3 Furnace and Charge Plate

As can be seen in Figure 4-1, the crucible is enclosed in a furnace section and has a charge plate attached to it. The assembly drawing in Figure 4-4 shows the entire upper section of the apparatus.

4.3.1 Furnace and Temperature Control

Once the crucible was designed the best heater had to be found that could provide enough heat to melt the metals. An embedded clamshell electric resistance heater was selected. The heater is made up of two semi-circles and provides 720 Watts of power running on 115 Volts. It is a Thermacraft 1200°C heater (Model RH 251). The heater has an inside diameter of 7.5 cm and an outside diameter of 10 centimeters. As shown in Figure 4-5, the entire heater is enclosed in ceramic fiber insulation with a one and a half inch diameter hole in the bottom to allow the spray to pass through into the spray chamber. The insulation package and furnace are enclosed in a cylindrical stainless steel section. The stainless steel section as shown in Figure 4-6 is a vacuum chamber. The section is made out of twenty-five centimeter outside diameter 304 stainless steel tubing and has two ISO Type-F vacuum flanges welded to it. The small lip at the bottom supports the insulation and the heaters. The entire furnace section was helium leak checked by the manufacturer to 10^{-11} torr.

The heaters are controlled by an Omega 9000A PID temperature controller. An Omega solid-state relay is used to switch the heater. The temperature is measured by an Inconel sheath type K thermocouple which is immersed in the molten copper. The thermocouple measurement feeds back into the controller which fires the solid state relay. The controller allows the adjustment of the proportional, derivative, and integral gains to fine tune the system response.

4.3.2 Charge Plate

The charge plate is made out of graphite, since graphite is a good electrical conductor and can withstand the high temperature. Another advantage of graphite is that the molten metal won't adhere to it. The graphite charge plate is electrically insulated from the crucible by a small ceramic spacer as shown in Figure 4-4. The spacer is glued to the crucible using Ceramabond 669 and the charge plate is adhered to the spacer. The charge plate has a three millimeter wide slot, which is lined up with the orifice. The charge plate is at a distance form the orifice, so that the jet breaks up into droplets, while it is passing through the slot in the charge plate. As is discussed in Section 2 the droplets all obtain an equal negative charge, and therefore repel each other in flight.

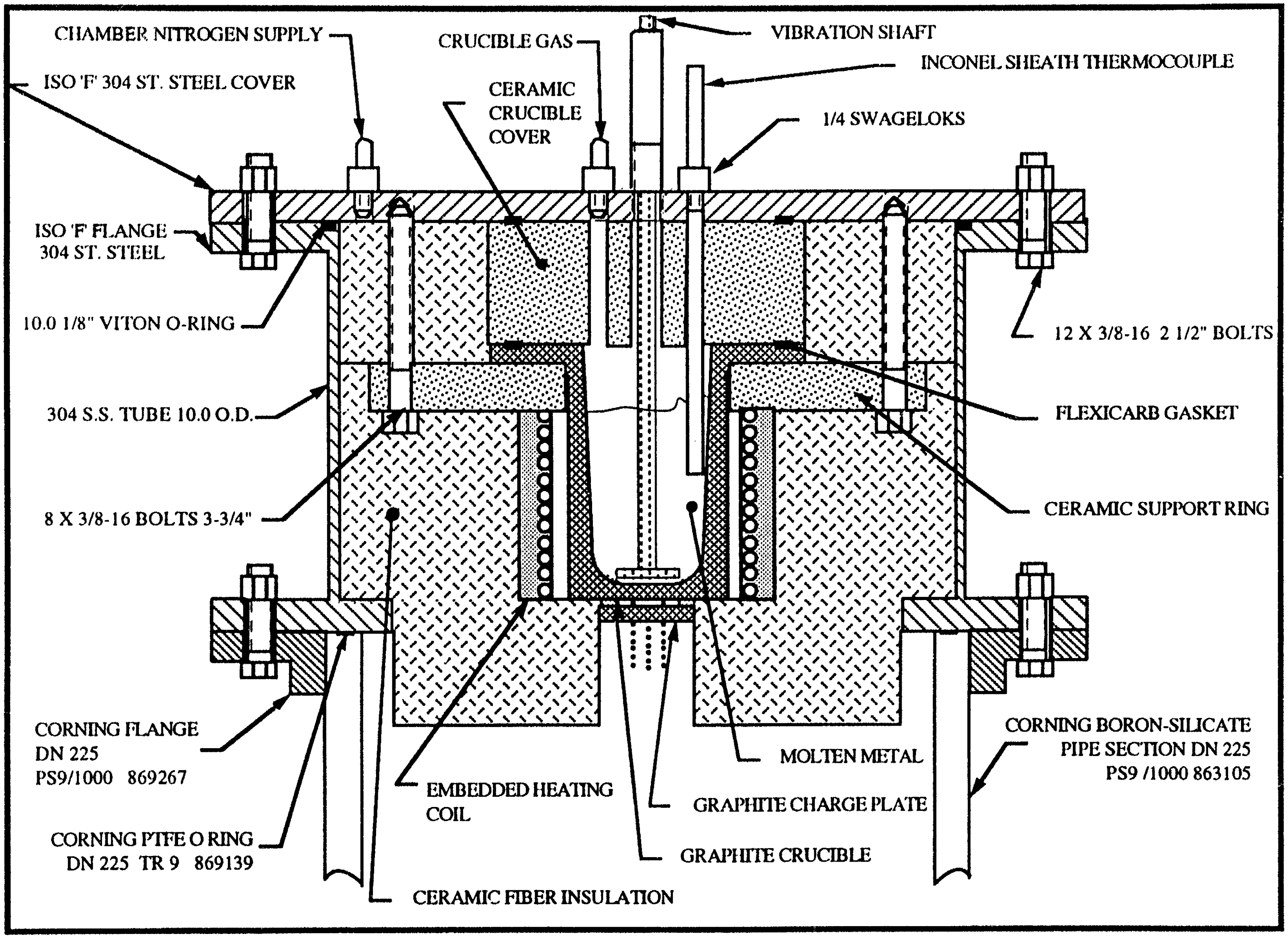

Figure 4-4: Assembly Drawing for Melt-Vibration-Charging System

4.4 Pressure Control and Spray Chamber

As can be seen in the assembly drawing in Figure 4-1 the apparatus has two different pressure regions. The entire system is sealed from the atmosphere and the crucible is sealed from the rest of the spray chamber and furnace. The furnace section is connected to a Corning Boro-Silicate glass spray Chamber (Model #DN225) which has an inside diameter of 23 centimeters. The glass chamber is designed for vacuums and positive gage pressures up to 80 KPa. The glass is also rated for temperatures up to 280° C.

4.4.1 Chamber Sealing

The system is sealed against the atmosphere in four different places. The furnace section is sealed against a stainless steel stop plate with a Viton O-ring as shown in Figure 4-4. A 3 mm (1/8") Viton O-ring is compressed by a twelve bolts between the furnace section flange and the top plate. The seal between the glass spray chamber and the furnace section is made using a PTFE O-Ring that is compressed by eight bolts which connect through a slip-on flange.

At the bottom of the spray chamber the glass is sealed against an aluminum base plate also using PTFE O-Ring and a slip-on flange with eight bolts as can be seen in Figure 4-5. The bottom of the spray chamber is sealed by an aluminum plate, which is bolted to the aluminum base plate. A 3mm (1/8") Viton O-Ring sits in an O-ring groove in the bottom plate and is compressed to seal the bottom of the chamber. Four clamps are used to hold the plate up during the assembly process.

There are also several feedthroughs in the chamber. In the top plate there are three electric power feedthroughs which seal to vacuum pressures of at least 10^{-6} torr. These provide power for the heater and the charge plate. There are also two feedthroughs to provide gas to the chamber and the crucible. There is one stainless steel nipple that allows the vibration shaft to go inside the crucible and one thermocouple feedthrough. The vacuum system is connected at the bottom of the spray chamber. The entire spray chamber is designed to allow a vacuum of at least 10^{-6} torr.

4.4.2 Crucible Sealing

The crucible is sealed from the furnace and spray chamber using two Flexicarb flexible graphite gaskets. One gasket seals between the crucible and crucible cover and the

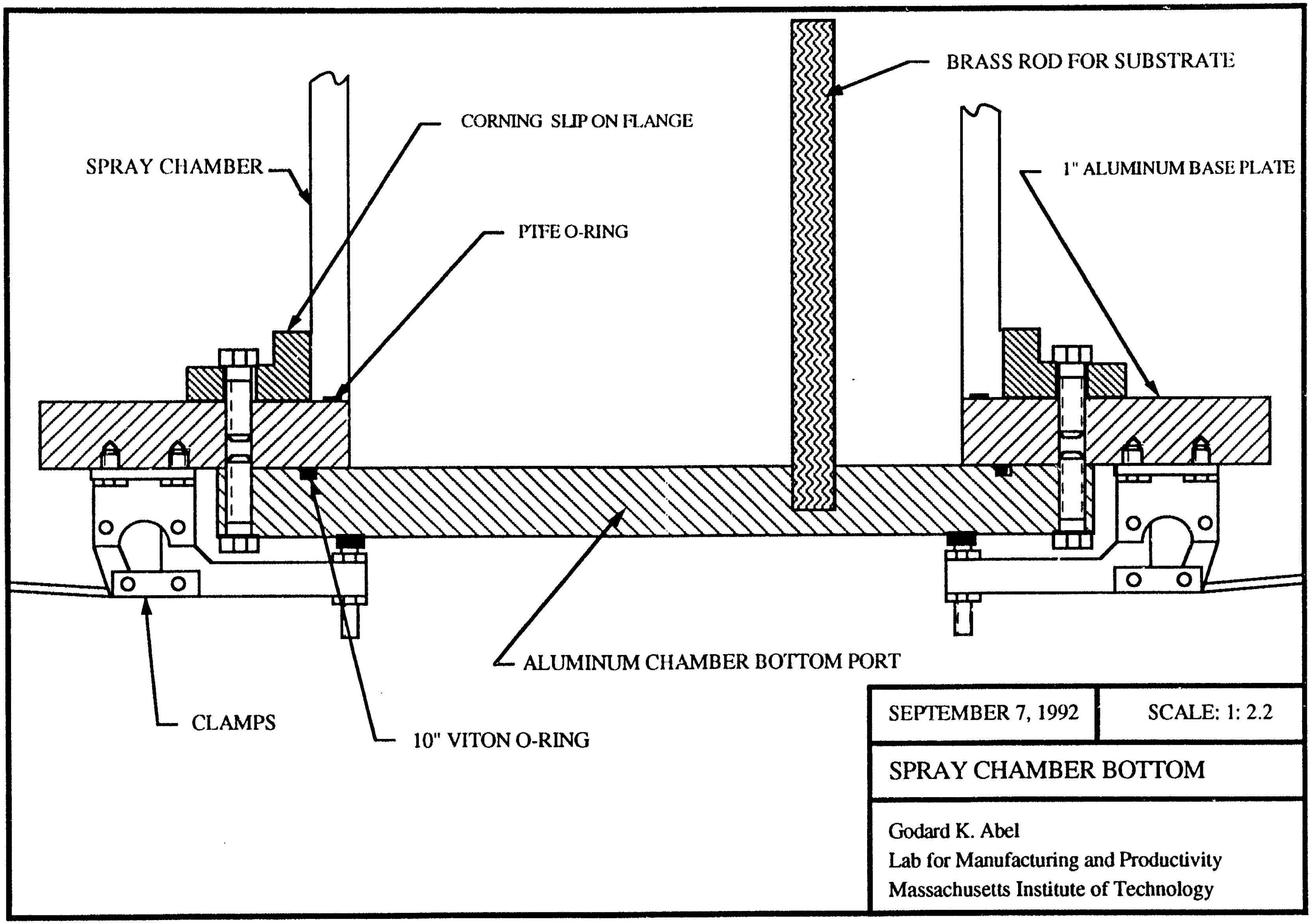

Figure 4-5: Bottom of Spray Chamber Assembly Drawing

other gasket seals between the top plate and crucible cover as shown in Figure 4-4. These gaskets are compressed by pressure applied from the support ring. The support ring is bolted to the top plate. The crucible is pressurized by nitrogen gas supplied through the top plate and the ceramic crucible cover.

4.4.3 Flushing the Spray Chamber

The chamber is evacuated from the bottom plate using a Welsch Scientific mechanical two-stage vacuum pump (Model #1376), which is capable of pumping down to 10^{-4} torr. Before each experiment the chamber is pumped down three times and re-filled with nitrogen to minimize the oxygen level in the chamber.

4.5 Piezo-Electric Vibration System

The piezo-electric vibration system uses a similar concept to the one used in the original system. However, the materials had to be modified to allow for the higher temperatures, a sealing lid was added, and the electrical connections were simplified. The piezo assembly is attached to the furnace section by a 3/8" NPT stainless steel nipple as shown in Figure 4-1. The piezo-electric transducer assembly is shown in detail in Figure 4-6. The vibration transfer shaft that goes into the crucible is made of an NZP zirconium based ceramic that has zero thermal expansion and a very low thermal conductivity. It also is non-wetting so the molten metal does not adhere to it. The piezo-crystal is a lead zirconate titanate made by Morgan Matroc (Model #PZT-5A).

The crystal is clamped between two brass pieces by an aluminum lid as shown in Figure 4-6. The sinusoidal input voltage is conducted to the crystal by a brass contact, which is connected by a wire connected through the lid. The brass contact is insulated from the rest of the assembly by a vernalic insulator piece. The rest of the assembly is grounded and is connected to the piezo-electric transducer through the lower brass plate. As the signal is fed into the piezo-crystal, it vibrates and creates an oscillation which is passed on to the melt through the shaft.

A 3mm (1/8") Viton O-ring seals the piezo assembly against the atmosphere and is clamped down between the aluminum base and cover with eight bolts. An eye bolt is attached to the aluminum cover and is used to lift the top plate onto the furnace section.

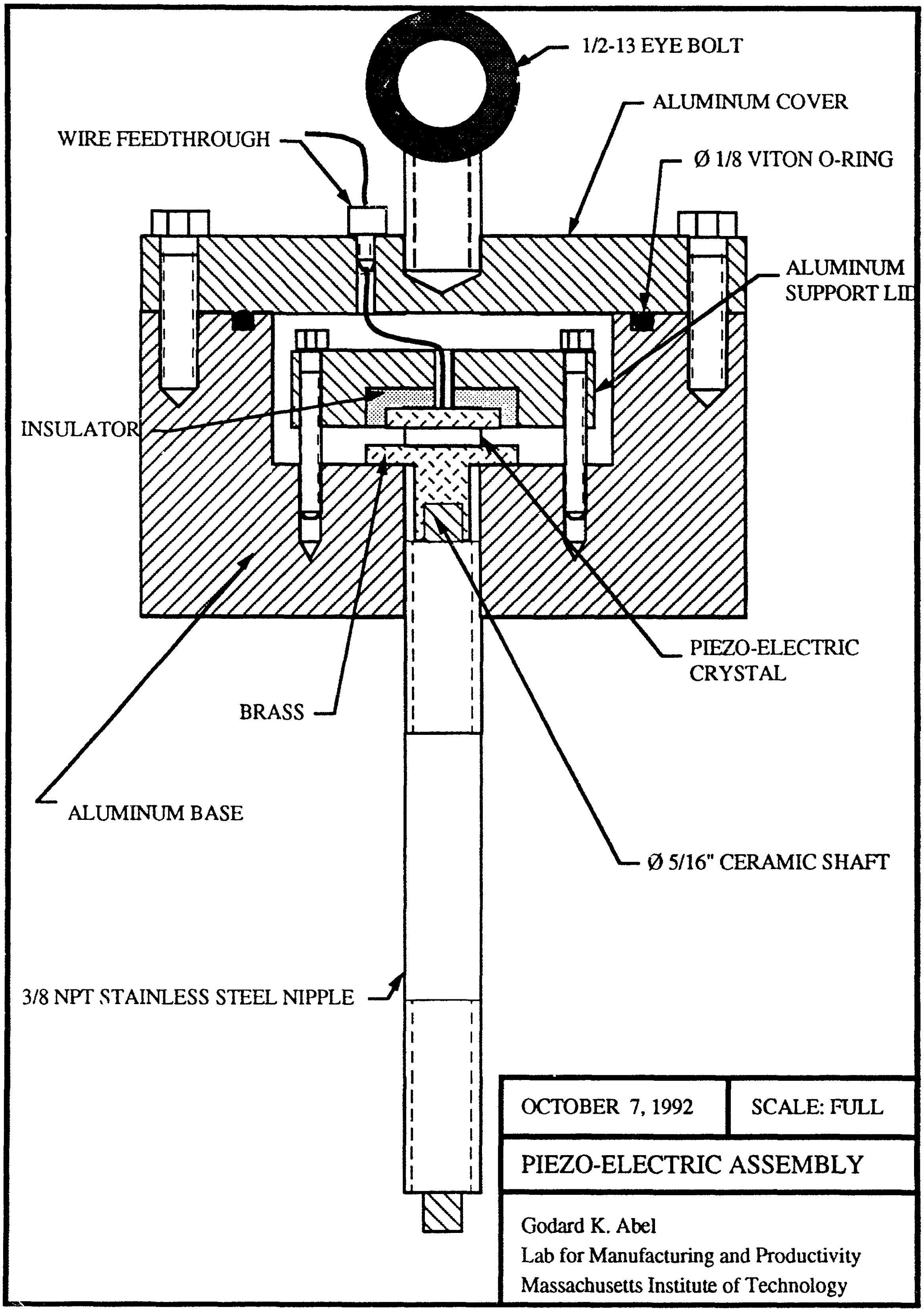

Figure 4-6: Piezo-Electric Vibration Mechanism

4.6 Substrate and Support Structure

4.6.1 Support Structure and Hoisting Mechanism

The entire assembly in shown in Figure 4-1 is supported by a Unistrut support frame. The aluminum base plate is bolted to the Unistrut support structure and supports the rest of the apparatus. A hoisting mechanism was also added to lift the top plate onto the furnace section. This was required since the top plate when it is completely assembled weighs approximately twenty kilograms. The hoist mechanism is made out of a winch that gives the operator a three-to-one mechanical advantage and has a ratchet lock that prevents the plate from falling if the winch is released. The winch is mounted to a roller that allows the plate to be rolled over the top of the furnace section and then be lowered into the chamber.

4.6.2 Substrate

The sprays can be collected on a substrate that can be located anywhere in the spray chamber by sliding a Pyrex disk up and down the brass pole located inside the spray chamber. The new spray chamber has a 23 cm inside diameter, which allows more room for larger substrates than the previous chamber, which only had a fifteen centimeter diameter. It is also one meter long which allows enough time for copper droplets to solidify before they reach the bottom of the spray chamber. For experiments where powder formation is desired a collector can be placed on the bottom of the chamber to collect the powder.

The entire furnace-vibration apparatus was designed to be modular so that it can be placed on different spray chambers. By detaching the furnace section from the glass chamber the entire furnace section can be moved to another spray chamber. Currently a large rectangular chamber is being built for spray coating and welding experiments, and the furnace system will be used with this spray chamber. It will be bolted to a mating flange on top of the rectangular spray chamber.

5. Results of Initial Testing and Design Modifications

Once the design, fabrication, and assembly of the new system were completed some initial testing was done to find potential problems in the system. Several problems were discovered and the design was then modified to correct these problems and optimize the performance of the system.

5.1 Furnace and Sealing Testing

The first test of the system was a simple heating experiment. The system was run in the open atmosphere, instead of a nitrogen environment, without a crucible and only a thermocouple in the furnace. This test was used to build up an oxide layer to on the clamshell heaters, since this extends their life, and it was also used to test the furnace and control system. The first test was successful as the furnace heated up to 1200° C in approximately half an hour. An additional thermocouple was placed at the outside of the ceramic fiber insulation between the support ring and the stainless steel cylinder. The ceramic fiber insulation kept the temperature at the outside of the insulation down to a maximum of 50° C.

The chamber sealing was also tested using an Illinois Instrument 2550 Oxygen analyzer. The oxygen level that was measured in the chamber during a heating experiment ranged form 25 to 35 parts per million of oxygen. The level of oxygen in the chamber remained low enough to prevent any visible oxidization of the graphite crucible during initial tests.

5.2 Fracture of Ceramic Support Ring

The first problem with the high temperature system occurred with the ceramic support ring. The holes in the ring were designed as loose clearance holes for the 3/8-16 bolts, however due to a fabrication error by the manufacturer of the NZP ceramic parts, the holes had a tight clearance. As the result of this error and a slight misalignment in the holes on the ring the tightening of the bolts applied radial stresses to the plate. When the bolts where tightened to seal the crucible, a fracture occurred in the ceramic ring. The manufacturer offered a free replacement part ; however, the manufacturer broke two of the plates while drilling the bolt circle. These problems with the ceramic ring led to a design modification.

5.3 Design Modification

The design was modified to reduce the stresses on the ceramic ring and to make it less expensive and easier to fabricate. A steel backup ring was added to the design as can be seen in Figure 5-1. The ceramic ring was made smaller and the bolts no longer go through the ceramic ring. This eliminates points of stress concentration on the ceramic ring, which served as crack growth sites for the brittle fracture. The strength requirement on the ceramic plate is also reduced since the distance between the crucible flange and the edge of the ceramic plate is reduced to 2.5 centimeters. This design change should make the ceramic support ring last much longer.

This design change also has disadvantages. The steel has a much higher thermal conductivity than the ceramic and this leads to more heat loss from the furnace and slows down the heating process. It also increases the temperature on the outside of the insulation where the wiring is fed into the chamber as shown in Figure 5-1 and increases the temperature of the entire stainless steel furnace section.

5.4 Melting Tests

Bronze powders with a melting point of approximately 1030° C, that were supplied by Alcan Powders and Pigments, NJ, were used for the initial testing. To test the melting procedure, a test was run where the crucible was not clamped to the top plate, but the ceramic ring and crucible were simply placed inside the furnace. Then the crucible lid and insulation were used to insulate the system. The chamber was flushed and filled with Nitrogen. The metal was heated past its melting point and to a maximum temperature of 1050°C in a period of approximately two hours.

The next step was to attempt to melt the metal and spray it through the orifice. This required the crucible to be clamped to the top plate. Since the new support ring from the manufacturer has not yet been received, the fractured plate was used with a steel backup ring. While tightening the bolts the ceramic plate was further fractured into four pieces; however, with the steel support ring the crucible could be firmly clamped and the Flexicarb gaskets were compressed. The introduction of the metal ring led to new problems. Since the steel ring conducted more heat to the outside of the stainless steel section, where the wires are led to the heater, the wiring now was exposed to much higher temperatures. This caused the insulation to melt off and led to short circuits.

This short-circuiting problem has been addressed. By replacing the plastic insulated wiring with wire that is protected by a ceramic overbraid the short circuits can be eliminated. One other way to simplify the wiring problem would be to weld a small nipple to the side of the stainless steel furnace section and then put two wire feedthroughs on the nipple. This would eliminate the problem of having to reconnect the wires for every experiment and make the system easier to use and more reliable.

5.5 Spraying Test

Once the short-circuiting problem was eliminated another test was performed to spray the bronze. The broken ceramic ring was used in combination with the steel support ring to clamp the crucible to the top plate, since the new ceramic ring has not arrived from the manufacturer. About 400 grams of bronze powder were melted and heated to a temperature of 1150° C in a period of two and a half hours. Once the metal was fully molten the crucible was pressurized to 275 KPa. The metal was sprayed through the 88μm orifice briefly and broke up into droplets of varying diameter, since no vibration was imposed on the melt. The orifice then was blown out of its pocket in the crucible, and the rest of the bronze was sprayed through the hole at the bottom of the crucible. The reason that the orifice was blown out is that the Aremco 668 ceramic adhesive that was being used did not adhere to the graphite well enough. The use of Aremco 669 Graphi- Bond should eliminate this problem. This test showed that the system could melt and produce bronze sprays. With the use of the vibration system, we should now be able to produce uniform droplets.

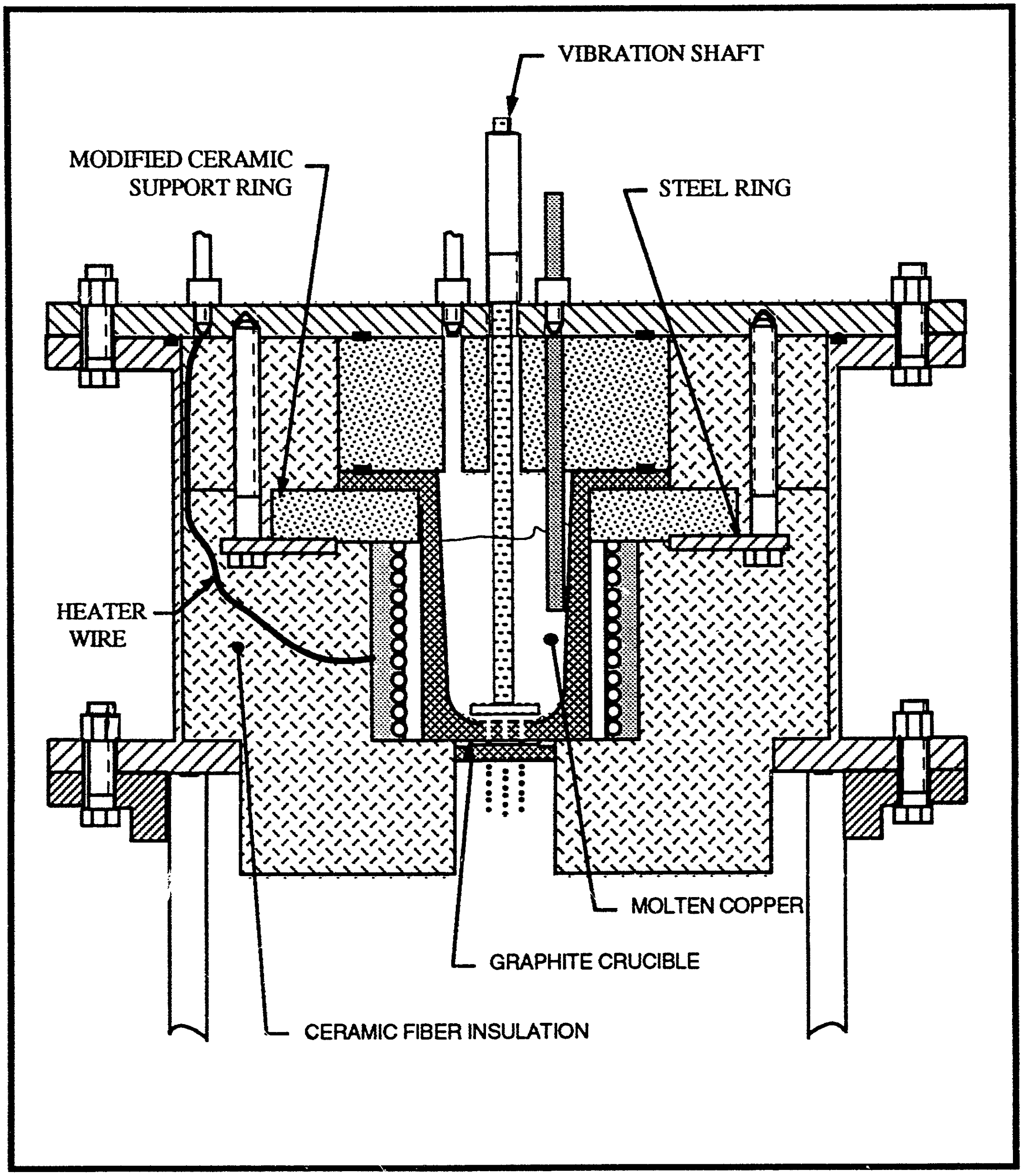

Figure 5-1: Modified Design of Furnace

6. Future Testing and Conclusions

With the design modifications outlined in the previous section the high temperature apparatus should be ready to produce uniform metal droplets. The initial objectives of testing will be to establish a consistent procedure for creating uniform droplet metal sprays and to begin charging the droplets. With the new apparatus we will be able to spray metals such as aluminum, copper and their alloys. Once we can consistently create uniform droplets then further characterization experiments can be run with the high temperature system.

The fundamental aspects of the DBM process such as the breakup mechanism and the charging mechanism can be studied for high temperature metals. Currently research is being conducted to understand the effects of process parameters, so as to develop scaling relationships for the DBM process. By determining the effects of the critical process parameters such as chamber oxygen level, charging voltage, vibration frequency, orifice diameter, and jet velocity, we will be able to create and control droplets with any desired thermal state for their deposition onto substrates.

Although the goal of creating uniform droplets with copper alloys has not yet been completely achieved, the high temperature system has been designed and constructed. By running some initial tests some modifications have been made to improve the system, and the ability to melt and spray high melting point metals has been shown. With the design modifications, we should be able to produce uniform droplet metal sprays consistently and characterize their properties. This will enable many applications of the DBM process to be developed.

References

Avallone, E.A. and T. Baumeister, eds. 1987. Marks' Standard Handbook of Mechanical Engineers. McGraw -Hill Book Company. New York.

Chun, Jung-Hoon and Passow, Christian H. 1991. "Spray Forming Process for Metal-Matrix Composites."

Crandall, Stephen H., Dahl, Norman C., and Lardner, Thomas J. 1978. An Introduction to the Mechanics of Solids. Mc-Graw Hill. New York.

Davies, John and Simpson, Peter. 1979. Induction Heating Handbook. Mc Graw Hill. London.

Lienhard, John H. 1987. A Heat Transfer Textbook. Prentice Hall Inc. Englewood Cliffs, N.J.

Megyesy, Eugene F. 1975. Pressure Vessel Handbook. Third Edition. Pressure Vessel Publishing Inc. Tulsa.

Passow, Christian Henry. 1992. "A Study of Spray Forming Using Uniform Metal Droplet Sprays." S.M. Thesis. Massachusetts Institute of Technology. Cambridge,MA.

Peterson, Rudolph Earl. 1974. Stress Concentration Factors. Wiley. New York.

Toyo Tanso Co. 1992. "Isotropic Graphite Engineering Data." Portland, OR.