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This paper presents an investigation of the power needs and impedance-matching techniques involved when heating small charges of relatively high-resistivity semiconductor materials. Topics of discussion also include a brief section on the theoretical basis for zone refining, relative merits of resistance and induction heating, travel mechanism requirements, and work coil design.

Considerable emphasis is placed on the efficiency of coupling between the induction heater and the load. Different methods of coupling the power into loads of silicon and germanium are indicated. The discussion on coupling centers around two work coil designs, a single-turn coil and impedance-matching transformer, and a five-turn coil, which is connected in series with the tank-circuit coil.

An induction heater capable of delivering 4 kw output power was found sufficient for melting a zone 2 cm long in a 1.5 cm diameter ingot. The system developed is capable of zone refining, zone leveling, or crystal growing with either silicon or germanium.

A LOW POWER ZONE-REFINING SYSTEM
FOR SEMICONDUCTOR MATERIALS

by

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LIST OF MOST FREQUENTLY USED ABBREVIATIONS

amps......................	amperes
BTU......................	British Thermal Unit
cm......................	centimeter
K......................	distribution coefficient
k......................	thermal conductivity
kcps......................	thousand cycles per second
kw......................	thousand watts
ma......................	milli ampere
Q......................	quality factor
M......................	mutual inductance
R......................	resistance
C......................	capacitance
L......................	inductance
w......................	radians per second
Δ......................	change in

A LOW POWER ZONE-REFINING SYSTEM FOR SEMICONDUCTOR MATERIALS

INTRODUCTION

The increasing demands for semiconductor materials of extremely high purity has encouraged the development and improvement of zone-refining techniques in recent years. Zone-refining facilitates the purification of material to previously unapproachable impurity levels, often less than 10 parts per billion. A need for silicon with less than one part impurity per billion exists because this pure material would aid in the evaluation of its intrinsic properties and lead to a better understanding of conduction phenomenon in high-resistivity semiconductors.

In addition to purification, zone melting has been successfully used for growing large, single crystals of semiconductors and for carefully controlling the impurity or doping concentrations. Zone leveling is a process whereby a specified doping may be uniformly spread with exacting accuracy throughout a crystal.

Most commercial zone refiners of semiconductors utilize radio-frequency power for heating the molten zone. The problem of coupling the load, a small ingot of rather high-resistivity semiconductor, to the generator often requires a specialized impedance-matching network. Efficiency of such a system is usually relatively low compared with the efficiency of coupling to a more desirable load, such as a steel shaft that might be surface hardened using induction heating. The usual approach to the problem of low efficiency is to use brute force;

therefore, most of the commercial induction-heating units have power ratings of at least 10 kw and range up to about 25 kw. With power levels of this kind, it is possible to have an extremely low, overall efficiency and still be able to successfully melt the charge.

The purpose of this report is to describe a zone-refining system for laboratory use which will utilize an induction heater of low-power capability. The power requirement situation is discussed along with what was found to be the most practical solution for this particular case. The investigation includes an analysis of the problems associated with both single- and multiple-turn work coils and their respective impedance-matching networks.

Results of this study include a workable system which is capable of melting silicon or germanium in a manner suitable for zone refining with a power output of less than 4 kw.

THEORY OF ZONE REFINING

Modern semiconductor devices require materials of extremely high purity and crystalline perfection. Studies of high-purity matter in many fields have shown that even the smallest measurable traces of impurities may exert a profound influence on the physical, mechanical, and chemical characteristics of these substances.

Zone refining is a further development of fractional crystallization, a process which has been known for many years and has been used commercially for purifying certain substances. In fractional crystallization an entire ingot of material is melted, then freezing is allowed to proceed slowly from one end of the ingot to the other. A given impurity may raise, lower, or have no effect on the melting point. If the impurity has no effect on the melting point, then no purification will take place. The overwhelming majority of impurities present in germanium and silicon do change the melting point and are segregated by the fractional-crystallization process. If a solute lowers the melting point of a solvent, its concentration in the freezing solid will be lower than in the liquid. Thus, solute will accumulate in the liquid. If the solute increases the melting point, the converse is true. Figure 1 is helpful in showing how this separation takes place.

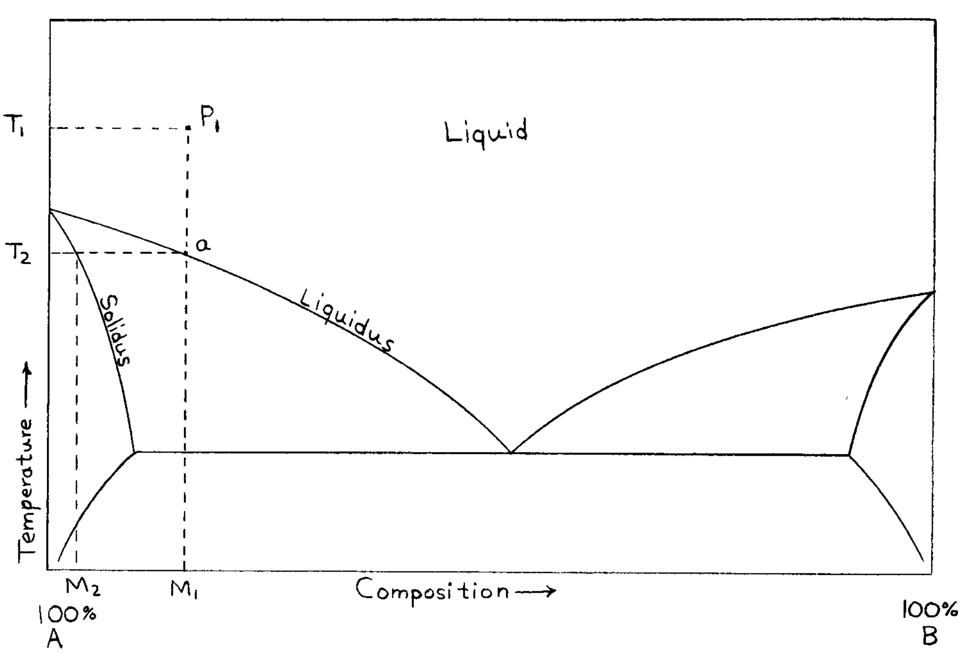


Figure 1. Phase Diagram for Two-element System A and B

Assume that the melt contains element A along with a single impurity B. The initial proportions are such that the mixture is M_1 on the abscissa and is at temperature T_1. Figure 1 shows that under these conditions the solution would be melted and completely mixed at point P_1. If the mixture is now allowed to cool, the point will move down the dotted line toward a. When the temperature of the cooler end of the ingot reaches T_2, part of the mixture will start to solidify, but at composition M_2 rather than M_1. Therefore, the end of the ingot which cools first will have a lower percentage of impurity B and will be purified.

The freezing interface will continue to move down the length of the ingot. Since more of element A than impurity B is freezing out, the melted portion will become enriched in B. Referring again to the phase diagram, one can see that this will result in an ever increasing percentage of B in the solid that freezes out. At some point the freezing solid will be of the same purity as the original mixture, and the last portion to solidify will have a much higher impurity concentration. The purity profile of an ingot that has been treated in this manner would appear as shown in Figure 2. Since no impurities are removed from the material, only redistributed, the area above the original impurity level is equal to the area below. This may not be obvious from the graph, because of the logarithmic scale used on the ordinate.

If further purification is desired, it is necessary to cut off and

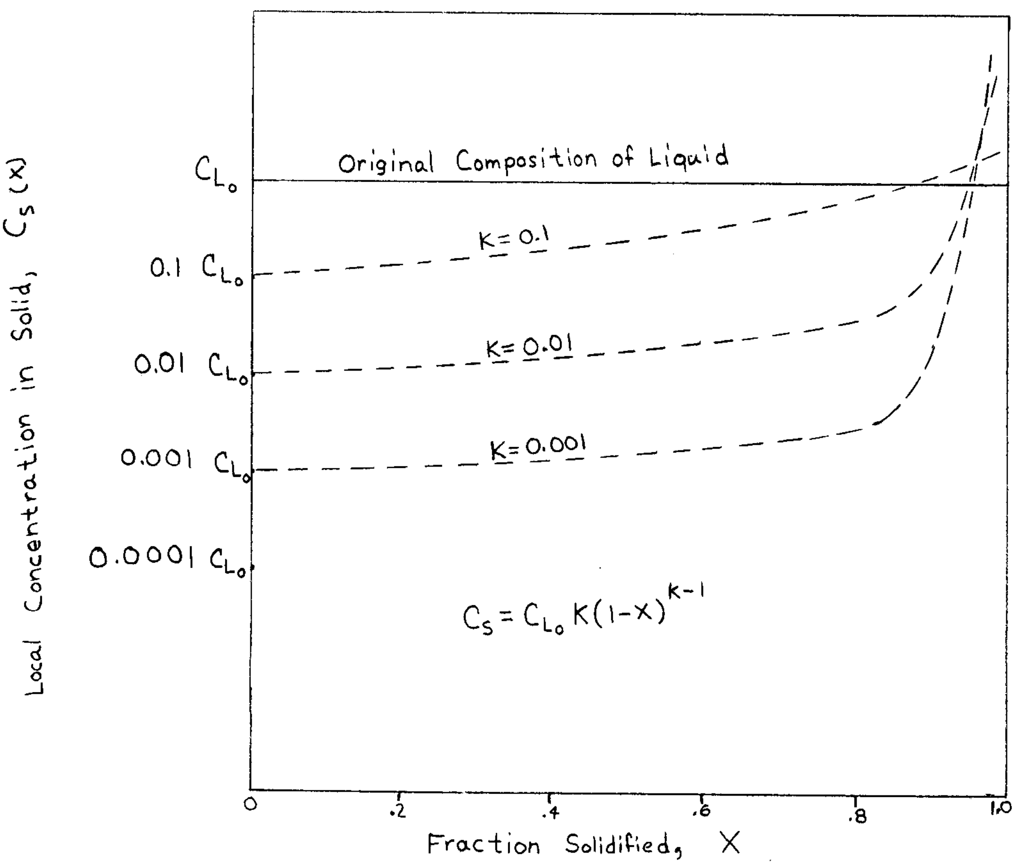


Figure 2. Impurity Concentration vs. Distance Along Ingot for Impurities with Various Distribution Coefficients (K)

(12, p. 455)

discard a portion of the impure end. The remaining piece is then remelted and the fractional-crystallization process repeated.

The ratio of percentage of solute, or impurity, in the liquid to the percentage impurity in the solid is defined as the distribution coefficient. The distribution coefficient (K) can be assumed constant for the small impurity levels dealt with in semiconductor work. This constant can be found in tables of distribution or segregation coefficients, from the appropriate phase diagrams, and from experiment.

The curve shown in Figure 2, page 5, can be plotted once the distribution coefficient is known. The formula

$$C_s = C_{L_o} K (1 - X)^{K-1}$$

will give this curve. C_s is the impurity level to solidify out, C_{L_o} is the initial impurity level in the melt, K is the distribution coefficient for the system, and X is the fraction of the total which has solidified (11, p. 455). It is apparent in Figure 2 that low values of K will result in good segregation, giving a lower impurity level for more than 90 percent of the bar length.

Zone refining relies on the same basic principle as fractional crystallization, except for one important difference. Rather than melt the whole ingot, only a narrow zone at one end is melted. This molten zone is then made to travel the length of the ingot. The zone has two liquid-solid interfaces, a freezing interface and a melting interface. At the melting interface, the solid material is being added to the molten zone in whatever proportion it happens to exist. At the freezing interface, however, the proportion of impurity to solidify will differ from the proportion in the melt according to the distribution coefficient. The distribution coefficient may vary from less than 10^{-3} to more than 10 (9, p. 3). In cases where K is greater than one, the purification will be in the opposite direction since the solid which freezes out of the melt will be less pure than the melt. In the remainder of this report K will be assumed less than one unless otherwise stated.

It is important that the zone travel slowly enough to allow diffusion of the impurities away from the moving interfaces to insure gaining the full benefit from the refining process. It is quite helpful to stir the melt because this tends to keep the zone homogeneous, rather than having a high impurity concentration at the freezing interface. The result of either too rapid a zone movement or insufficient melt agitation will move the effective value of K closer to one.

The advantage of zone refining over fractional crystallization is that many successive zones may be passed through the charge, each decreasing the impurity level without having to cut off the impure end. Provided the charge is long enough, it is even possible to have more than one zone moving through a charge at the same time.

Calculations for impurity level as a function of distance along the ingot become more complicated than in the fractional-crystallization case because the additional variable, zone length to ingot length must be considered. Normally, curves such as those in the appendix of Zone Melting by W. G. Pfann are used for computing the impurity concentration. These curves show the relative solute concentration, C/C_0, as a function of distance along the ingot, the distribution coefficient, the zone length to ingot length ratio, and the number of passes.

ELEMENTS OF A ZONE-REFINING SYSTEM

A system for zone refining consists of two basic components. First, there must be a device which can hold the molten charge in place and provide motion for the repeated passes necessary for the refining process. This device, or "travel mechanism," should provide a wide range of highly-stable speeds so that accurate work with a variety of charges can be accommodated.

The zone travel must always be in the same direction, which means that the travel mechanism will have to have a rapid return to the beginning point between each pass. If possible, this should be incorporated into an automatic system which can be set to work unattended. Some provision must be made for supplying an inert atmosphere to the charge since the elevated temperature necessary for melting would also cause excessive oxidation and contamination.

It is desirable to have some method for stirring the melt to minimize the building up of impurities at the freezing interface. The more vigorously the melt is stirred, the faster the allowable zone-travel speed, thus resulting in a more efficient, overall system.

The charge can be contained in an open crucible or boat. This boat should be made of some material that will not contaminate the molten charge or be in danger of melting or breaking. Alternate methods exist, such as the "floating zone method," but a discussion of such techniques is beyond the scope of this paper (9).

The second major component of the zone-refining system is the heat source. Several factors should be taken into consideration when choosing a method for heating the charge. Since the molten zone requires an inert atmosphere, a means for simultaneously heating and excluding air must be devised. The heat source must not add to the contamination of the charge.

Generally, zone length will be quite short. There is no single formula for ideal zone length, inasmuch as several variables are involved. Some compromise between usable ingot length and purity is necessary. The most commonly-used choice for zone length is from .1 to .2 times the ingot length. In any case, the zone boundary should be as sharp as possible, and the zone length should be highly stable.

Power requirements will be dictated by the nature of the material to be refined and the physical size of the proposed zone. Most commercial zone-refining systems use induction heating. Although little information is available as to the minimum-power rating of the heaters, most units in use are from 10 kw to 25 kw. In the next section, an estimate will be made of the power required to melt zones of the size that will be demanded of this zone-refining system.

ESTIMATION OF POWER REQUIREMENTS

The zone-refining system should be capable of zone leveling and crystal-growing operations as well as purification. The maximum ingot diameter that the heater is capable of melting should be at least 1.5 cm in order to give the device reasonable versatility. If we assume a zone length of 2 cm, the areas involved can be calculated.

Area of cross section (both ends):

$$A = 2\pi r^2 = 3.53 \text{ cm}^2$$

Area of surface:

$$A = (2 \text{ cm})\ (\pi)\ (1.5 \text{ cm}) = 9.42 \text{ cm}^2.$$

Some heat will be lost through conduction to the solid portion of the ingot and some through convection and radiation from the zone surface. Allowing several simplifying assumptions, this loss may be calculated. See Appendix IV for details of this calculation. The estimated loss for the zone which is 1.5 cm diameter by 2 cm long is 840 watts. The power source must be able to supply at least 840 watts to the load to maintain the molten zone.

THEORETICAL COUPLING EFFICIENCY

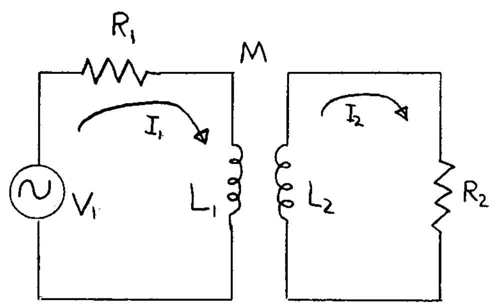
Two logical possibilities exist for heating sources that are compatible with the available travel mechanism. These sources are a resistance heater and an induction heater. The resistance heater could probably be used to some satisfaction, but since the heat is generated outside the quartz tube and radiated toward the charge to melt it, the zone length would be long and the zone interfaces poorly defined. Silicon melts at 1426^{o}C and because the quartz tube would be closer to the resistance coils than to the charge, the tube would get considerably hotter than this. Excessive heat could melt the tube and would add to the chance of the container contaminating the molten silicon.

Although the equipment needed is bulky and expensive, the use of induction heating is generally accepted as the best means of heating for zone refining of semiconductors. The heat is generated within the charge itself. This means that the container and the tube will both be at a lower temperature than the melt, minimizing the contamination. The stirring effect that the eddy currents have on the melted material is important in that it eliminates the necessity of providing mechanical stirring. Any length coil can be used to provide an easy means of controlling zone length. In materials such as silicon and germanium, which are relatively poor thermal conductors, the zone boundaries are quite sharp. Also, zone length can be controlled by changing the power input to the coil.

The energy that can be dissipated in the charge will be a function of the power rating of the induction heater and the overall efficiency attained. The theoretical power requirement has been estimated at 840 watts. Solving for the theoretical coupling efficiency will give an idea of the induction heater power rating necessary to deliver this required power.

Regardless of the type of work coil or the coupling network used, the generator-load system can be represented by the simplified equivalent circuit shown in Figure 3.

Figure 3. Equivalent Circuits of Generator and Load.



In the equivalent circuit, V_1 represents the oscillator voltage, R_1 the resistive, and L_1 the reactive portion of the impedance of the work coil. L_2 represents the inductance of the charge placed in the work coil and R_2 the effective value of resistance. I_2 is the current which flows in the load and causes the heating. The load would be analogous to the short-circuited secondary of a transformer; thus, the circuit relations would be:

$$V_1 = R_1 + (jwL_1)\ I_1 + jwMI_2$$

and

$$0 = jwMI_1 + (R_2 + jwL_2)\ I_2.$$

Solving for the impedance presented to the oscillator gives:

$$\frac{V_1}{I_1} = R_1 + jwL_1 + \frac{(wM)^2}{R_2 + jwL_2}$$

$$= R_1 + \frac{(wM)^2 R_2}{R_2^{\ 2} + (wL_2)^2} + jw\left[L_1 - \frac{(wM)^2\ L_2}{R_2^{\ 2} + (wL_2)^2}\right].$$

The effective value of primary resistance has been increased and the inductance decreased by the load placed in the work coil. If we define Q as $\frac{wL_2}{R_2}$

then the change in the resistance and inductance, respectively, is:

$$\Delta R_1 = \left(\frac{M}{L_2}\right)^2 R_2 \cdot \frac{Q^2}{Q^2 + 1}$$

and $$\Delta L_1 = -\left(\frac{M}{L_2}\right)^2 L_2 \cdot \frac{Q^2}{Q^2 + 1}.$$

The efficiency of the entire circuit will now be:

$$\text{Efficiency} = \frac{\Delta R_1}{R_1 + \Delta R_1}$$

$$= \frac{\left(\frac{M}{L_2}\right)^2 R_2 \cdot \frac{Q^2}{Q^2 + 1}}{R_1 + \left(\frac{M}{L_2}\right)^2 \cdot R_2 \ \frac{Q^2}{Q^2 + 1}}.$$

Q will nearly always be at least 3, which means that $\frac{Q^2}{Q^2 + 1}$ will approach 1. Making this assumption, we have finally:

$$\text{Efficiency} = \frac{\left(\frac{M}{L_2}\right)^2 \frac{R_2}{R_1}}{1 + \left(\frac{M}{L_2}\right)^2 \frac{R_2}{R_1}} .$$

Frequency does not enter into the above formula, and therefore does not influence the efficiency. It is true that skin effect does play an important role at the frequencies commonly used, but since both the load and the circuit coils will be affected the same, the ratio of $\frac{R_2}{R_1}$ will remain constant (2, p. 91-93).

This expression for the efficiency, though difficult to evaluate in this form, shows which parameters are important and may serve as a guide in perfecting an efficient power transfer. Since the resistivity of molten silicon or germanium is much higher than that of copper, the ratio $\frac{R_2}{R_1}$ should be large. However, it is less apparent whether $\left(\frac{M}{L_2}\right)^2$ will be big or small, and since this term is squared, it will have a greater influence on the overall efficiency than the resistive term. This indicates that the load diameter should be large to reduce L_2 and the spacing close between the work coil and the load to increase the mutual inductance.

A useful relationship exists for the case of a conductor parallel to a flat conducting sheet. The conducting sheet, if placed near the conductor, will be in the magnetic field of the conductor and will have heating currents induced in it. The efficiency of this heating can be theoretically derived and is found to be (8, 119-121):

$$\text{Efficiency} = \frac{1}{1 + \frac{h}{a}\sqrt{\frac{\sigma_m}{\mu_m \sigma_c}}} \qquad \text{where}$$

h is the distance from the center of the conductor to the surface of the sheet, a is the radius of the conductor, σ_m is the conductivity of the load, μ_m is the relative permeability of the load, and σ_c is the conductivity of the conductor.

This expression gives an approximation of the efficiency of a single-turn coil surrounding a cylindrical load. This formula was used to find the efficiency of a single-turn 1.5 inch diameter coil loaded with a 1.5 cm silicon ingot. The result was approximately 59 percent efficiency. (Refer to Appendix I)

This efficiency gives only the losses incurred in coupling from the work coil to the load, and does not include losses elsewhere in the tank circuit. A matching transformer, in which further losses are incurred, has to be included in the circuit when using a single-turn coil. This is necessary since the loosely coupled single-turn coil presents a very low impedance and must be transformed to a high value.

The estimated loss in the matching transformer and tank circuit is about 35 percent, making the total efficiency figure .59 x .65 = 38 percent. (See Appendix II) Successful operation would require a sizable power surplus to overcome factors such as heat of fusion and long initial melting time.

The theoretical efficiency figures arrived at assume ideal conditions and, therefore, would be maximum values. In all probability, the values actually experienced would be slightly smaller due to imperfections in manufacture of the circuit.

AVAILABLE EQUIPMENT

There was a class C power oscillator available in the Electrical Engineering Department that had been built in 1950. It was thought that this unit might be adapted for use as a heat supply in the proposed zone-refining system. Power was provided by three Eimac 304 TL's working in parallel. The circuit was designed for a maximum plate voltage of 1500 v and a maximum d-c plate current of 2 amps, for a total input power of 3000 watts. Assuming 67 percent efficiency (reasonable for a class C oscillator), this leaves a usable output of 2100 watts for heating purposes. Maximum allowable plate dissipation is 300 watts per tube for a total of 900 watts. Therefore, this would be a maximum-power design for this voltage level.

A theoretical coupling efficiency of 38 percent means that of the 2100 watts output power, only 800 watts will be delivered to the load. This is less than the power calculated for melting the zone, and so the induction heater will not be satisfactory for the zone-refining system in its present form. The next section discusses modifications that were made to the induction heater.

At the present time, there is available a travel mechanism especially for zone refining built by Tektronix Incorporated. This device has been loaned by Tektronix to the Department of Electrical Engineering at Oregon State University.

The travel mechanism has a d-c motor-driven carriage with continuously variable speeds from one inch per day to five inches per minute. The speed control has a mechanical transmission which gives anywhere from a 1:1 to a 1000:1 ratio in nine discrete positions. The d-c motor can then be run at speeds from about 500 rpm to 4000 rpm. A tachometer shows the motor speed, and this, along with the transmission ratio, is used with a chart to find the exact travel speed. The machine may be programmed to run from one to nine cycles and then shut off, or it can be set to run continously until shut off manually. The return speed is independent of the forward-travel speed but can be changed if necessary. The return mechanism is powered by an a-c motor. Changing the speed can be accomplished by using different sized drive pulleys. The machine is designed so that either the charge or the heat source may move while the other remains stationary. The charge is placed in a small boat which in turn is put inside a length of quartz tubing. The quartz tube can be capped and some inert gas passed through the tube to insure a non-contaminating atmosphere. This travel mechanism is satisfactory for the proposed zone-refining system.

REDESIGN OF INDUCTION HEATER

The power-limiting factor in the original oscillator was the operating point chosen for the tubes. Complete tube data, obtained from Eitel-McCullogh Corporation, showed that the tubes could be used with plate voltage as high as 3000 volts. This gives 1200 watts output power per tube when plate current is 500 ma. This rating again gives 300 watts plate dissipation, which is the maximum value for continuous operation.

A thorough investigation of the entire circuit showed that the next highest-stressed component after the oscillator tubes was the d-c supply. The rectifier tubes, 872 A's, are designed to deliver 1250 ma d-c, or peak currents of 5000 ma. This means a maximum d-c plate current of 2.5 amps may be drawn from the d-c supply.

The maximum d-c voltage under load was found to be slightly more than 2500 volts. At 2500 volts the maximum plate current should be about 550 ma. This indicates that the tubes would not be utilizing all the available current from the rectifier tubes. An investigation of the filiment transformer showed that it was working at about 65 percent of rated current and could easily heat another tube. A fourth tube was placed in the induction heater in parallel with the original three. The total plate current was now 2200 ma which is reasonably close to the rated output of the d-c power supply. The design voltage was 2500 volts which takes full advantage of all the voltage available. The net output power was raised from 2100 watts to about 4100 watts by redesigning the circuit operating points and adding a fourth tube. See Appendix III. Assuming the same efficiency as before, the power delivered to the load would be .59 x .65 x 4128 = 1580 watts, which is well over the estimated power required to melt a zone. Appendix V shows a simplified circuit diagram of the induction heater as revised.

There are several critical variables in a class C amplifier or oscillator which must be properly adjusted in order to obtain optimum performance. The high efficiency of the class C oscillator is a result of the fact that plate current only flows for a small part of each cycle, when the instantaneous plate voltage is low. The d-c grid bias and the peak r-f grid voltage will determine θ, the number of electrical degrees the plate current will flow. Any design one uses will be a compromise because, for a given circumstance, a larger value of θ will result in greater output current while a smaller value of θ will give a higher plate efficiency. As this oscillator is to be used in a laboratory zone-refining system, total output power was the primary design criterion with the limitation of 300 watts plate dissipation per tube. Plate current flows for about 150° in this design, giving an efficiency of 78 percent.

Load impedance is not a basic parameter, but rather is a function of the desired plate-voltage swing, peak-plate current, and the d-c plate bias E_b. The output power per tube can be expressed in terms of these same parameters which give an evaluation of the peak a-c plate current, I_1 (14, p. 462).

$$\text{Power Output} = \frac{(E_b - E_{min})\ I_1}{2}$$

E_b is the d-c bias and E_{min} is the minimum instantaneous plate voltage. E_{min} is usually chosen to be some value slightly greater than E_{max}, the maximum instantaneous grid voltage. Since E_g = -350 volts and the peak r-f grid input voltage is 520 volts, E_{max} will be 170 volts. A convenient value for E_{min} is 200 volts. This value gives a satisfactory compromise between output power and efficiency.

Then:

$$\text{Power Output} = 1080 = \frac{(2500 - 200)\ I_1}{2}$$

$$I_1 = \frac{2(1080)}{2300}$$

$$= .94 \text{ amps.}$$

When the proper load impedance is placed in series with the plate circuit, the plate-current pulses will generate the desired alternating plate voltage. The expression for finding the load impedance is (14, p. 462):

$$\text{Load Impedance} = \frac{E_b - E_{min}}{I_1}$$

$$= \frac{2500 - 200}{.94}$$

$$= 2450 \text{ ohms.}$$

In the induction heater, four identical tubes are placed in parallel. Therefore, the desired load would be 612 ohms. This figure can be used as a guide and should give results very close to the calculated values of current and power. The load impedance at the work coil will be much smaller than 600 ohms, but using the proper impedance-transforming network should change the low impedance to the required value.

The circuit uses grid-leak bias, which results in a self-stabilizing oscillator. The value of the grid resistor is found by dividing the desired d-c bias by the average, or d-c value, of the grid current. This is:

$$R = \frac{350 \text{ volts}}{332 \text{ ma}}$$

$$= 1060 \text{ ohms.}$$

The grid current is the total grid current for the four tubes. Power rating for the grid resistor must be at least

$$P = I^2 R$$

$$= (332 \text{ ma})^2 \, 1060$$

$$= 117 \text{ watts.}$$

Observation of circuit performance may indicate that a change in grid bias would be desirable. This may be easily accomplished by increasing or decreasing the grid-leak resistor. The a-c grid input voltage may be varied by changing the turns on the grid pick-up coil or by changing the coupling between the grid coil and the tank coil. The adjustment will have to be made experimentally since only a rough approximation may be made theoretically.

FREQUENCY EFFECTS

Earlier in the report it was shown that the efficiency of the power transfer from the coil to the load is not affected by frequency, inasmuch as the effective resistance of the charge and the coil increase a like amount as the frequency is raised. However, frequency may have an influence on the amount of power a charge of a given diameter will absorb from an r-f field.

For a cylindrical charge of uniform cross section in a helical coil, the approximate power dissipated per square centimeter is (3, p. 456):

$$P = H^2 \ (\rho \mu f)^{1/2} \ /8\pi$$

where ρ = resistivity in emu = 10^9 x resistivity in ohm-cm, μ = relative permeability ≈ 1 for silicon and germanium, f = frequency in cycles per second, and H = 4π NI, the magnetic field strength.

An optimum frequency, defined as the frequency above which no gain in power dissipation may be realized, can be derived from the above expression and is:

$$f \ (opt) = 6.25 \ \rho/8\pi^2 \ \ r_o^{\ 2}$$

where r_o is the radius of the work piece in centimeters. The resistivity of molten silicon or silicon very near the melting point is not known exactly, but it has been estimated at between .01 ohm-cm and .001 ohm-cm (3, p. 456). The formula assumes that the load nearly fills the coil -- that is, the coupling is very tight. Applying this expression for an ingot 1 cm in diameter, we find that the optimum frequency is:

$$f\ (opt) = 6.25\ \rho / 8\pi^2 (1cm)^2$$

$$= 7.91 \times 10^4\ cps$$

if we assume ρ to be .001 ohm-cm or 7.91×10^5 cps if we assume ρ to be .01. Because it is necessary to have a quartz tube between the coil and the workpiece, the coupling will be much looser than the formula assumes. This would indicate that a higher frequency would be beneficial for increasing the efficiency. As the diameter of the workpiece becomes small in comparison to the diameter of the coil, it becomes difficult to mathematically define an optimum frequency. Experimental trials with different frequencies seem to be the most satisfactory method for finding the frequency which will give the best overall performance. An effective frequency in preliminary lab work was found to be 350 kcps. This is a commonly-used frequency in commercial zone refining of semiconductors. The tubes in the oscillator are rated for full power up to 30 mc so that frequency may be increased later if desired.

DESIGN OF WORK COILS

The approximate load impedance that the oscillator is designed to work into is 600 ohms. Before an impedance-changing circuit can be designed, it is necessary to know the impedance at the terminals of the work coils. The book Induction Heating by N. R. Stansel, a 1949 McGraw Hill publication, devotes an entire chapter to analytical methods for calculating the impedance of loaded and unloaded coils. These methods may be used with a minimum of effort because all the hard-to-evaluate terms are in graph form. Direct measurement was used for all but the preliminary work in this paper, but the analytical method is convenient if the necessary equipment for direct measurement is not available.

Coil impedance measurements were made on a General Radio type 916A Bridge which will measure accurately down to about .1 ohm at all frequencies of interest. Since it was impossible to have molten silicon for a test load, carbon, with a resistivity of about .008 ohm-cm was substituted. Several coils of varying sizes and shapes were wound, using copper tubing of 1/4 inch, 5/16 inch, and 3/8 inch outside diameter. The quartz tube to be used in zone refining had a 1-1/2 inch outside diameter; therefore, all coils were wound to give a loose fit on the tube and to allow travel of the zone. Early experiments showed that the 3/8-inch tubing was almost impossible to bend to a 3/4-inch radius, even after annealing. Either 1/4-inch or 5/16-inch tubing could be used satisfactorily, but 5/16 inch was chosen since it would have less resistance. Table I shows input impedances for three coils at 350 kcps as measured by the General Radio 916A Bridge.

If R_L is the resistive component of the loaded coil and R_u is the resistive component of the unloaded coil, the ratio of $\frac{R_L}{R_L + R_u}$ gives the maximum efficiency that could be achieved using the

Table I. Impedance Values of Work Coils at 350 kcps Measured With General Radio 916A Bridge.

Coil	Unloaded		Loaded	
	R	+jX	R	+jX
Single turn with 16:1 matching transformer	.1 ohm	12 ohm	.25 ohm	10 ohm
Five-turn helical	.16	3	.43	2.4
Five-turn double cone	.15	3	.4	2.5

particular coil and load. The simulated load represents the condition after the silicon has reached melting temperature. Initiating heating in the silicon is another problem which will be discussed in a later paragraph.

COUPLING TO SILICON LOAD

A parallel-resonant circuit is the ideal load circuit to use with a power oscillator since it may be used for transforming the load impedance to the proper value as well as determining the oscillating frequency. The coils and capacitors used in the tank circuit of an induction heater must be of sufficient rating to handle the voltage and current loads placed on them. Tank current will be approximately Q times the plate current.

Common values for loaded tank Q's range between 10 and 20, although both higher and lower Q's may be quite satisfactory. Unloaded Q, designated Q_u, should be as high as possible, inasmuch as the overall efficiency of the loading circuit is

$1 - \frac{Q_L}{Q_u}$. High values of Q_L offer greater discrimination against harmonics and facilitate matching to lower impedance work coils, but tank currents will be high resulting in increased losses in the tank coil.

The work coil may be placed in parallel with the tank coil, in series with it, or inductively coupled by means of an air-core transformer. Another possibility is to put the work coil in parallel with a portion of the tank coil to get an autotransformer effect. The method used will depend on the desired load impedance for the oscillator and the actual impedance of the loaded work coil. If these values are equal or nearly equal, parallel or autotransformer connection will be appropriate. If the work-coil impedance is much lower, then series connection can be used. For extreme differences, where a factor of several hundred is involved, inductive coupling and an additional matching transformer may be necessary.

In designing the tank circuit for any particular work coil and frequency for the zone-refining system, coil choice was virtually unrestricted since all coils were hand wound and could be made to desired specifications. Capacitors, however, presented a problem since hand fabrication was not practical and the supply of factory-made capacitors of the required ratings was very limited. The lack of capacitors can be partially made up by allowing the frequency to vary over a wide range rather than specifying a particular frequency. Thus, when C has been designated, L may be chosen to give the desired Q rather than some exact frequency.

Specific examples using the work coils listed in Table I will help show how the desired load impedance may be achieved using the low impedance work coils and a matching network. The two coils, which will be discussed, are the five-turn double cone and the single-turn coil. Both of these coils were found to give very short zones, which is important when processing ingots of limited overall length. The tightly wound five-turn coil was tapered in toward the middle from both ends and gave a much shorter zone than the five-turn helical coil, but the impedance value was nearly the same. The single-turn coil gave an even shorter zone, but the impedance was very low and this made matching a more difficult problem. Figure 4 shows a picture of the single-turn work coil and matching transformer. Figure 5 shows the five-turn work coil.

The five-turn coil may be connected directly in series with the tank coil. This eliminates the losses associated with a matching transformer, but tank losses may be higher because the same current which flows through the work coil must also flow through the tank circuit. The series impedance of the loaded work coil and the tank coil combine to give an equivalent parallel impedance that the oscillator sees (1, p. 72):

$$Rp = R_s \ (1 + Q_s^{\ 2}) \qquad \text{where } Q_s = \frac{X_s}{R_s} \ .$$

From Table I, R_s = .5 ohm; therefore, setting Rp = 600, we find that Q_s must be 35. R_s includes .4 ohms from the loaded work

Figure 4. Single-turn Work Coil and Matching Transformer.

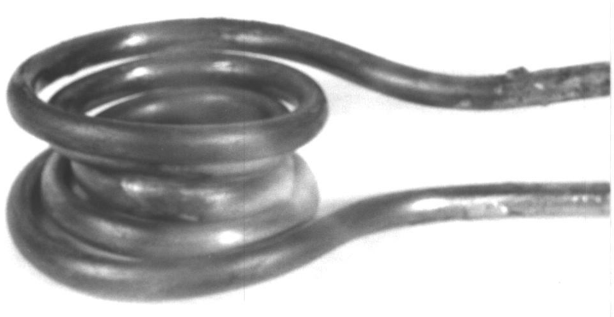


Figure 5. Five-turn Work Coil.

coil and approximately .1 ohms from the rest of the tank circuit.

It can be shown that the series Q will be very nearly the same as the parallel Q for all cases where $Q_s \geqslant 5$. Since

$$Q_p = \frac{R_p}{X_p}, \quad Q_s = \frac{X_s}{R_s}, \quad X_p = X_s \left(1 + \frac{1}{Q_s{}^2}\right), \text{ and}$$

$$R_p = R_s \left(1 + Q_s{}^2\right) \text{ and assuming } Q_s \geqslant 5$$

$$Q_p = \frac{R_p}{Xp} \approx \frac{R_s\, Q_s{}^2}{X_s} = \frac{Q_s{}^2}{Q_s} = Q_s .$$

If $Q_p = 35$, then $\frac{600}{X_p} = 35$. Therefore, X_p must be 17 ohms. A .03μf capacitor is available and this will resonate with a 7μh inductor and give X_p= 17ohms at 340 kcps. These values are approximate, but close enough for a first trial. This combination was tested using the dummy load and gave excellent results.

The exact operating characteristics of the oscillator were monitored using a Tektronix type 516 dual-trace oscilloscope with 10:1 attenuating probes. This allowed simultaneous display of plate and grid voltage waveforms up to a peak-to-peak value of 1200 volts. The limit of 1200 volts did not allow operation of the unit at full design levels, but extrapolation may be used with some confidence, as long as the Q of the circuit does not change excessively with increased loading.

The oscilloscope allowed proper adjustment of the peak grid voltage by changing the number of turns in the grid pickup coil. The minimum plate voltage, a function of load impedance, was found to be very close to the design point. This is important since an excessively high E_{min} will give decreased plate efficiency. A value of E_{min}, which is less than E_{max}, the maximum instantaneous grid voltage, will result in high grid currents due to secondary emission from the plate.

An alternate method of checking circuit performance is by observing d-c grid current, d-c plate current, and d-c bias voltage on the plate. Proper loading and circuit adjustment will result in rated plate and grid currents flowing when the maximum plate voltage is applied. An easy method for finding the approximate overall efficiency is to compare the plate current under loaded and unloaded conditions where

$$\text{Efficiency} = 1 - \frac{I_u}{I_L} .$$

This does not give the exact efficiency since the operating conditions do not remain the same, causing the plate and grid dissipation to change.

Efficiency of the induction heater and five-turn work coil using this method is:

$$E = 1 - \frac{.1}{.22}$$

$$= 55 \text{ percent.}$$

This is higher than the theoretical efficiency calculated for the original three-tube unit because the test load is of larger diameter, 2 cm, than the 1.5 cm minimum charge diameter previously assumed.

Expected zone length with the five-turn coil falls within the acceptable limits. The carbon test block could be made to glow white hot for varying percentages of its length, depending on the power applied. Power transfer to the load is easily controlled by regulating the d-c supply voltage. Small pieces of silicon were placed on a carbon block which was placed inside the coil. When power was applied, the silicon melted, confirming the fact that high enough temperatures can be reached using the induction heater.

The resistivity of silicon changes two or more orders of magnitude as it is heated from room temperature to its melting point. The resistivity vs temperature curve, however, is positive from room temperature up to some elevated temperature, then it becomes steeply negative to the melting point. Therefore, when the heating process first starts, the load resistivity is much higher than the designed value, and as the specimen starts heating, this mismatch becomes worse. The result is that there is not enough power absorbed by the load to heat it more than a few hundred degrees. It is necessary to use some other means to initiate heating beyond the point where the resistivity reaches a maximum and starts to decrease. No exact data were taken as to the precise temperature at which the silicon will self-heat since this depends on the doping and would vary among samples. It appears that the silicon must be heated to a temperature of about 1200°C in most cases before self-heating takes effect.

The initial heating can be accomplished in several ways. One method successfully used was to place the silicon in a small carbon boat. The carbon was heated by the induction heater and this in turn heated the silicon to the critical temperature. When the critical temperature is reached, power absorption from the r-f field exceeds losses and the material continues heating until the melting point is reached. Since carbon and silicon react at high temperatures, contamination from the carbon boat would occur, proving this method undesirable. The carbon is needed only for the initial heating, and so this suggests another method which would be better for processing an ingot.

A band of tungsten or molybdenum may be wound around one end of the ingot. This will heat the portion of the ingot directly under the band, and as the work coil is moved down the ingot, conduction will preheat the silicon and insure a proper impedance match for the length of the ingot.

No experiment was conducted where a zone was moved through an entire ingot as described above, but experiments in which large carbon blocks were maintained at temperatures above the melting point of silicon proved that the 4 kw induction heater has the power needed to process ingots at least 1.5 cm in diameter.

The single-turn coil listed in Table I was silver soldered onto mounting plates which bolt to the output terminals of a matching transformer. The primary of the output transformer consists of a 16-turn coil wound with 5/16-inch copper tubing and is in series with the tank coil. The secondary is a single turn of heavy copper sheet, which gives a minimum resistance to the secondary circuit. The entire transformer is water cooled.

Table I shows that the real part of the input impedance of the transformer is .25 ohms when the coil is loaded. Thus, the required tank circuit Q is 49 and X_L and X_c at resonance should be 12.3 ohms. Since the only available capacitor is again .03 μf, L will have to be 4 μh, which makes the frequency of oscillation about 450 kcps. Slightly higher values of impedance could be expected at 450 kcps than the values measured at 350 kcps, but this may be neglected in the first trial.

Efficiency of the system using the matching transformer and single-turn coil, while lower than that for the five-turn coil, was still quite satisfying. Plate current increased about 60 percent when the load was placed in the coil, indicating an efficiency of

$$E = 1 - \frac{1.25}{2}$$
$$= 37.5 \text{ percent.}$$

It is worthwhile to discuss what effect changing the turns ratio of the matching transformer would have on the power losses.

Assume, for instance, that the number of turns was increased until the impedance reflected into the tank circuit from the single-turn coil was doubled. This would require about 1.4 times as many turns in the primary coil and would increase the resistance of the primary coil by 1.4 since the resistance is a function of the conductor length. The resistance reflected into the tank circuit has now been doubled and this will mean that the tank Q should be less by a factor of .707. This assumes that the resistance of the main tank coil and the change in inductance of the primary coil are both small compared with the total tank-circuit resistance and inductance. The tank current, Q times the plate current, will be less in the same proportion, and therefore, the tank I^2R losses will be reduced. This may be carried only so far because the transformer efficiency will drop off as the turns ratio is increased due to leakage flux. Efficiency of the system could doubtless be improved by going to a ratio of about 25:1.

The success of the five-turn coil with its associated high efficiency and short zone length has made it the choice for the zone-refining system as it now stands. A single-turn coil, or a two-turn coil adapted to fit the matching transformer, is available and may be used if future demands on the system change.

COUPLING TO GERMANIUM LOAD

The melting point of germanium is 940°C, nearly 400°C lower than the melting point of silicon. Conduction loss is directly proportional to temperature and radiation loss is proportional to the fourth power of temperature. Assuming the same zone size as before, 1.5 cm diameter by 2 cm long, the total heat loss would be approximately one-half of the loss encountered with silicon.

The resistivity of molten germanium is about .003 ohm-cm (12, p. 18). This figure is comparable to the resistivity estimated for molten silicon, which means that the coupling charactertistics of the two materials are similar. Only about one-half of the available power output of the oscillator is needed to maintain a molten zone, but the load impedance must be reasonably well matched to the tubes to prevent excessive plate or grid dissipation. The d-c plate bias can then be regulated to control energy input to the load, which in turn will determine the zone length.

Germanium may be melted in either carbon or ceramic boats without excessive contamination from the boat material. Carbon makes an ideal boat material because it will preheat the germanium to the point where it will absorb heat directly from the r-f field. If a ceramic material is used, then it becomes necessary to devise some other method of preheating, as was the case with silicon. The same methods will apply, except that the critical temperature is much lower with germanium.

An experiment was conducted to test the effectiveness of the heating unit with the five-turn work coil for melting germanium. A piece of germanium 1.75 cm long was cut off the end of a single-crystal ingot of 40 ohm-cm germanium. The ingot was 1.5 cm diameter, resulting in a volume of 3.6 cc. A small ceramic boat was used as a mold to form a new ingot 5 cm long and approximately 1 cm in diameter. Heating was initiated by placing a small piece of carbon next to the germanium. The germanium proved to be a much easier material than silicon to preheat. The carbon block was removed once the germanium started to absorb power from the field, allowing the germanium to flow freely down into the boat.

The diameter of the original germanium ingot was about the same as in the carbon test load which was used for designing the impedance matching network. When the material melted down into the boat, however, the diameter was reduced and this affected the impedance match. A new coil was wound, similar to the five-turn coil but with 11 turns. The inductance of this coil was 3 μh compared with 1 μh for the five-turn coil, which required a corresponding reduction in the tank-coil inductance.

The maximum power that could be delivered to the load under these circumstances was 1500 watts, enough to melt a zone over 4 cm long. The material in the center of this zone was much hotter than necessary. If a zone length this long was desirable, a helical coil could be designed that would maintain a more even heat distribution and give higher efficiency.

Very short zones could be melted in the germanium. An input power of 250-350 watts was sufficient for melting a zone of about 1 cm. Zone length is very difficult to measure, but having a stable zone is much more important than knowing the exact length of the zone.

The heater unit has more than enough power for the zone refining of germanium. Matching is not critical as long as the load is not so far from the design load that excessive plate dissipation becomes a problem due to low operating efficiency. A visual check on the color of the plates will usually provide ample warning.

CONCLUSION

When this project was started, there were several objectives. One was to determine how much power would be required for a zone-refining system capable of melting silicon ingots 1.5 cm in diameter. Another was to find the coupling efficiency that could be expected from an induction heater.

Figures for the power requirement and coupling efficiency of an induction heater loaded with such a charge were calculated. When these figures were compared with the output of the available induction heater, it could be seen that the heating unit was too small.

The redesign of the induction heater was successful in that it provided the additional power necessary for melting the silicon. The power of the heater after alteration, 4100 watts, is still much lower than most commercial units. A large power surplus is advantageous since this eliminates the need for careful impedance matching but this report points out the fact that smaller units can be used.

The objective of developing a working zone-refining system was fulfilled. The present combination works very well for processing germanium as well as silicon. In addition to zone refining, the system can be used for zone leveling and crystal growing. This equipment will be used as an experimental tool by the solid state technology class, a graduate-level class in the Department of Electrical Engineering at Oregon State University.

Some additional equipment may be purchased in the future which will greatly extend the usefulness of the zone-refining system. An extra tank-circuit capacitor of about .005 μf would give increased flexibility to the frequency range and impedance-matching capabilities. Carbon boats could be purchased or manufactured for processing germanium. A worthwhile project for the future would be to adapt the present system to perform floating-zone refining of silicon.

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APPENDICES

APPENDIX I. Proof of Theoretical Coupling Efficiency From Single-Turn Coil to Load.

Assume that the effective conductivity of silicon is .001. This would include the molten silicon of the zone and some solid silicon on each side of the zone (3, p. 456). Assume 5/16 inch outside diameter water cooled copper tubing conductor.

$h \approx$ 5/8 inch

$a \approx$ 5/32 inch

To find σ_c, skin effect must be considered. Define d as reference depth where a hollow tube with wall thickness d has a d-c resistance equal to the a-c resistance of a solid, cylindrical conductor of the same radius as the tube (15, p. 103).

$$d = 3160\sqrt{\frac{\rho dc}{f}}$$

$$= 3160\sqrt{\frac{68 \times 10^{-6}}{350 \times 10^{3}}}$$

$$= 4.43 \times 10^{-3}$$

$$\frac{R_{ac}}{R_{dc}} = \frac{1}{4}\left(\frac{a_o}{d} + 1\right)$$

$$= \frac{1}{4}\left(\frac{.313}{4.43} \times 10^{-3} + 1\right)$$

$$\doteq 18$$

$$\rho_{dc} = .68 \times 10^{-6} \text{ ohm-in} = 1.7 \times 10^{-6} \text{ ohm-cm}$$

$$\rho_{350 \text{ kcps}} = (18)(.68 \times 10^{-6}) \text{ ohm-cm}$$

$$= 30.6 \text{ ohm-cm}$$

$$\sigma_m = \frac{1}{.001} = 1000 \text{ mho}$$

$$c = \frac{1}{.306} \times 10^{-4} = 3.36 \times 10^{4} \text{ mho}$$

$$\text{Efficiency} = \frac{1}{1 + \frac{h}{a} \sqrt{\frac{1000}{3.36 \times 10^{4}}}}$$

$$= \frac{1}{1 + 4\left(\sqrt{\frac{1}{33.6}}\right)}$$

$$= 59 \text{ percent}$$

APPENDIX II. Proof of Losses in Matching Transformer and Tank Circuit.

The best method for measuring the losses in a water-cooled circuit is to determine the heat absorbed by the water. Since the conductors will be kept at low temperatures by the cooling water, the radiation and convection losses will be negligible, thus, the method should be very accurate. Two tests were made, one with the single-turn coil and matching transformer, and the other with the five-turn work coil in series with the tank coil. In the first case, the water was run through the work coil, the matching transformer, and the tank coil. In the second case, no matching transformer was used, so the water was run through the work coil and tank coil only. Results are shown below.

Case 1 - Single-turn coil with matching transformer.

Power output of oscillator - 1000 watts

Water flow rate - 6.56 cc/sec

Temperature of water in - $20^{\circ}C$

Temperature of water out - $33^{\circ}C$

Since 69.7 gram-calories per minute = 1 kw,

$$\text{Loss} = 69.7 \times 60 \times 85.3 \times 10^{-3}$$

$$= 356 \text{ watts.}$$

Case 2 - Five-turn coil in series with tank coil.

Power output of oscillator - 1000 watts

Water flow rate - 6.56 cc/sec

Temperature of water in - $20^{o}C$

Temperature of water out - $30.5^{o}C$

Loss = $69.7 \times 60 \times 69 \times 10^{-3}$

= 315 watts

APPENDIX III. Design Specifications for Power Tubes in Induction Heater.

Design Specifications -- Four 304 TL's in Parallel.

	Each tube	Total
DC Plate voltage	2500 v	2500 v
DC Plate current	550 ma	2200 ma
DC Grid voltage	-350 v	-350 v
DC Grid current	83 ma	332 ma
Peak RF Grid input voltage	520 v	520 v
Driving power	38 watts	152 watts
Grid dissipation	10 watts	40 watts
Plate power input	1380 watts	5520 watts
Plate dissipation	300 watts	1200 watts
Plate power out	1080 watts	4320 watts
Net power out		4128 watts

APPENDIX IV. Proof of Heat Loss From Molten Zone of Silicon

The heat losses from the molten zone will be broken down into two parts. First is the combined loss due to radiation and convection from the zone. Conduction to the adjoining parts of the ingot constitutes the second loss median.

Curves are available which show the combined radiation and convection losses from a surface as a function of temperature and the emissivity of the surface. The Brown Boveri Review gives such a curve (11). Assuming the emissivity to be near one, the curve shows a loss of about 60 watts per cm^2 at $1430^{o}C$, for a total radiation and convection loss of 9.42 cm^2 x 60 watts/cm^2 = 565 watts.

Conduction loss is much harder to evaluate because the exact value of the coefficient of thermal conductivity of silicon at high temperature is not known. Another unknown factor is the temperature profile beyond the molten boundaries. If enough assumptions are allowed, however, a figure can be obtained which may be accurate to ± 20 percent.

The expression for conduction into an infinitly long cylinder of cross-sectional area A, perimeter P, and temperature difference T_s - T_∞ between the heated end and the cool end is (6, p. 47):

$$q = \sqrt{\bar{h}\ P\ k\ A}\ \ (T_s - T_\infty).$$

In this formula, k is the coefficient of thermal conductivity and $\bar{h}$ is a factor which gives an average value for the convection and radiation losses along the surface of the cylinder. Near the molten zone the radiation would be important and toward the cool end of the cylinder convection would be the major loss. This factor was estimated at $\frac{4\ \text{BTU}}{(\text{hr})(^{\circ}\text{F})(\text{ft})}$ with the advice of M. B. Larson, Associate Professor of Mechanical Engineering at Oregon State University. $T_s - T_\infty$ would be the difference between the zone temperature and the ambient.

The coefficient of thermal conductivity of silicon at room temperature is $\frac{49\ \text{BTU}}{(\text{hr})(^{\circ}\text{F})(\text{ft}^2)}$. No available reference listed a value of k for high temperatures, although it was noted that for semiconductors the value of k generally decreased with increasing temperatures. Using these estimated parameters and converting to the proper units, it can be shown that q = 470 BTU/hr or conduction loss = 138 watts since the loss will be the same on both ends of the ingot, total loss = 276 watts. Total loss from radiation, convection, and conduction = 565 + 276 = 841 watts.

APPENDIX V. Simplified Circuit Diagram of Redesigned 4 kw Induction Heater.