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GROWTH OF HIGH PURITY OXYGEN-FREE SILICON BY COLD CRUCIBLE TECH--ETC(U)

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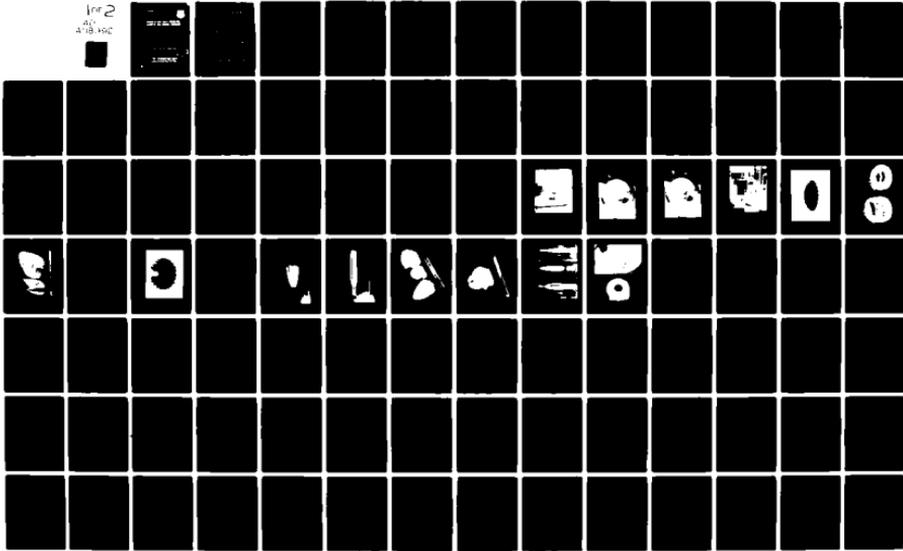
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Final Technical Report
June 1962

GROWTH OF HIGH PURITY OXYGEN FREE SILICON BY COLD CRUCIBLE TECHNIQUE

Ceres Corporation

Joseph F. Wendrus
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ried out on the thermodynamics and heat flow characteristics within the melt confined in the cold crucible in an effort to develop a better understanding of the crystal growth process.

The goals of the program have been achieved. Specifically, we have completed the following tasks:

- An extensive literature/patent search on RF induction melting and cold crucible technology, particularly as it relates to the production of high purity silicon, has been completed. The resultant bibliography which includes several hundred relevant journal articles and patents has been appended to this report.
- A water cooled copper cold crucible (73 mm I.D.) which incorporates a bottom-feeding mechanism, was designed, constructed and tested successfully.
- Silicon melts (weighing 750 grams) were routinely melted using low frequency induction heating (~ 250-300 KHz) and confined within the cold crucible for extended periods.
- Seeding techniques were developed to permit the Cz growth of single crystals of silicon with lengths up to 100 mm and diameters in excess of 25 mm.
- Representative samples of the single crystals grown from melts confined in the cold crucible (as well as samples of the poly-silicon feed material used) were submitted to the RADC Technical Monitor for characterization and analysis. While there are inconsistencies in the results of the various groups which characterized the material, it can be concluded that:
 - a) There is no evidence of silicon melt contamination resulting from extended periods of melt confinement within the cold crucible.
 - b) All crystals grown from melts confined in the cold crucible exhibit an unusually low oxygen content - typically 1 PPM or less.
 - c) The crystals grown exhibited a high level of carbon contamination (2-30 PPM) which we believe, is caused by the use of a graphite radiator/reflector assembly which was employed to optimize the seed/melt interface temperature.
 - d) Typically, the dislocation densities of the as-grown silicon crystals were found to be in the range of $3 \times 10^3/\text{cm}^2$ to $4 \times 10^4/\text{cm}^2$. However, it is most encouraging to note that one of the crystals evaluated to date was found to be dislocation free.

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SUMMARY

The two basic methods used to manufacture all of the single crystals silicon used today in the semiconductor industry are the Czochralski (Cz) method and the Floating Zone (FZ) technique. The Cz method, i.e. crystal pulling from melts contained in quartz crucibles, is the most widely used process at the present time; over 1,000,000 kg of single crystal silicon are produced annually using this process. While CZ-grown crystals exhibit a high degree of crystallographic perfection i.e. dislocation-free crystals are now manufactured on a routine basis, crystal purity, notably oxygen contamination caused by the quartz crucible, is a major problem which impacts upon device performance and yield.

The Floating-Zone (FZ) method, which utilizes high frequency induction heating techniques to produce and maintain a molten zone of silicon without a container, is the only other process used today for large scale silicon crystal production; approximately 200,000 kg of FZ silicon are produced annually. While the purity of FZ silicon crystals is significantly better than Cz silicon crystals (the oxygen impurity level is less), the typical crystal perfection achieved is limited to a great extent by the thermal geometry inherent to the FZ process. Moreover, the FZ crystal diameter which can be produced is constrained by the volume of molten silicon which can be supported by surface tension combined with RF levitation effects.

Over the past two (2) decades a continuing search has been underway to develop improved, low-cost methods for the production of high purity silicon crystals; ideally, a process which would combine the crystal perfection of CZ crystal production with the purity levels achieved by the containerless FZ techniques. The cold crucible/skull melting technique in which a molten column of silicon is inductively melted and confined within a water-cooled cold crucible structure appears to be a

practical combination of the best features of the Cz and FZ methods. Crucible contamination is eliminated since the molten column of silicon is confined by the RF field without physically contacting the walls of the cold crucible. The base of the molten silicon column is supported on the unmelted portion of the silicon feed material so that the melt is not in contact with any contaminating solid. Moreover, the volume of the melt which can be stably maintained within the cold crucible is not limited by surface tension effects as in the FZ process.

The goal of the program was to explore the feasibility of utilizing a cold crucible system for the growth of high purity, oxygen-free single crystals of silicon. The work included a detailed evaluation of previous research on cold crucible assemblies and the investigation of the growth of single crystals of high purity silicon utilizing the water-cooled cold crucible. In parallel with the experimental work, a theoretical analysis was carried out on the thermodynamics and RF field interaction with the melt confined in the cold crucible in an effort to develop a better understanding of the crystal growth process.

The goals of the program have been achieved. Specifically, we have completed the following tasks:

- An extensive literature/patent search on RF induction melting and cold crucible technology, particularly as it relates to the production of high purity silicon, has been completed. The resultant bibliography which includes several hundred relevant journal articles and patents has been appended to this report.
- A water-cooled copper, cold crucible (73 mm I.D.) which incorporates a bottom-feeding mechanism, was designed, constructed and tested successfully.
- Silicon melts (weighing 750 grams) were routinely melted using low frequency induction heating (~ 250-300 KHz) and confined within the cold crucible for extended periods.

- Seeding techniques were developed to permit the Cz growth of single crystals of silicon with lengths up to 100 mm and diameters in excess of 25 mm.

- Representative samples of the single crystals grown from melts confined in the cold crucible (as well as samples of the poly-silicon feed material used) were submitted to the RADC Technical Monitor for characterization and analysis. While there are inconsistencies in the results of the various groups which characterized the material, it can be concluded that:
 - a.) There is no evidence of silicon melt contamination resulting from extended periods of melt confinement within the cold crucible.

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1.0 INTRODUCTION

Silicon is the major semiconductor material used today for device manufacture in the solid state electronics industry. Production of silicon has reached substantial proportions--world-wide output of polycrystalline silicon (the raw material used for subsequent crystal growing operations) exceeded 1,800,000 kg in 1980 and increased to over 2,400,000 kg in 1981 to meet anticipated production needs (Arthur D. Little, Inc. estimates).

Over the past twenty years, silicon crystal production methods have been steadily developed to provide improved yields, higher purity and lower costs. However, there is growing evidence that further refinement of existing technology or the continued expansion of production capacity utilizing existing production methods, has begun to show diminishing returns in response to the increasingly stringent demands for crystal purity and perfection.

While the production of polycrystalline silicon has become a highly-sophisticated, automated chemical process industry producing tonnage quantities of product with impurity levels in the parts-per-billion range, the production methods used to manufacture single crystal silicon have not kept pace. Indeed, the principal crystal production methods (Czochralski and Floating-Zone techniques) remain batch operations using essentially the same technology developed over 25 years ago - although greatly expanded to meet the increasing capacity needs of the semiconductor industry.

The two basic methods used to manufacture all of the single crystal silicon used today in the semiconductor industry are the Czochralski (Cz) method and the Floating-Zone (FZ) technique. The Cz method, which is the most widely used process at the present time, involves the growth of a silicon crystal (via vertical-pulling) from molten silicon which is contained in a fused quartz crucible. Over 1,000,000 kg of single crystal (up to 125 mm diam.) are grown annually via this process utilizing sophisticated Cz furnace systems which contain silicon melts weighing up to 60 kg. Automatic crystal diameter/melt temperature controls are used to produce high yields

of silicon crystal which are virtually dislocation-free. Unfortunately, silicon melts react with the quartz container and Cz-grown silicon crystals are found to contain a significant quantity of impurities such as oxygen which necessitates further processing to reduce its electrical activity.

The Floating-Zone (FZ) method is the only other process used today for large-scale silicon crystal production; approximately 100,000 to 200,000 kg of FZ crystal with diameters up to 100 mm are produced annually. In the FZ process, high frequency induction heating is used to melt a portion of a polycrystalline silicon feed rod. The molten zone which is axially-supported by the surface tension of the melt, is seeded (as in the Cz method) and by moving the molten zone away from the seed/melt interface, controlled nucleation and crystal growth is allowed to take place.

While the FZ process permits the production of silicon crystal which contain less oxygen impurity, striations are more pronounced in FZ crystals because the thermal conditions in FZ furnaces are less symmetrical than those typically found in Cz pullers.

Over the past two decades, a continuing search has been underway to develop improved, low cost methods for the production of high purity silicon crystals. Containerless methods, similar to the FZ process, have been pursued as a means of improving crystal purity. Levitation methods have also been explored in which the silicon is not only melted by RF induction heating, but also physically supported by the RF field. While this melting technique has proven to be an intriguing concept, the weight of the silicon melt which can be fully levitated by the RF field is severely limited, thus restricting the process to research applications.

Cold hearth melting and casting of refractory metals using induction heating methods were described in the early 1960's. In 1966, researchers at TOCCO-STEL (France), a manufacturer of induction heating equipment, were the first to successfully adapt a skull melting/cold crucible approach to the fusion of silicon. Using this technique, the molten column of silicon which

is confined in a water-cooled cold-crucible structure is squeezed radially and contained by the RF field. The base of the molten column is supported on the unmelted portion of the feed material; indeed, the melt is physically supported but is not in contact with any contaminating solid.

A French patent was granted on this process (U.S. Patent applications were never filed) and other than the patent document, details of this work were never published, nor to our knowledge pursued further.

In 1977, with internal funding, researchers at Ceres Corporation initiated an intensive program on the adaptation of cold-crucible technology to the fusion and recrystallization of high purity silicon. Using a cold-crucible assembly of proprietary design, Ceres' researchers succeeded in pulling poly-crystalline silicon ingots from melts confined in the cold-crucible. Results of Ceres' preliminary experiments demonstrated that this new technique could be used to maintain large, stable melts of silicon with minimal, if any, oxygen contamination.

During the course of this program, techniques have now been established for the Czochralski growth of high purity single crystals of silicon from melts confined in a cold crucible. Characterization of representative samples indicates that with the exception of carbon, the purity level of the silicon single crystals produced have been significantly enhanced as a result of the cold crucible melting and the crystal growth process. The analytical data shows quite clearly, that the cold crucible Czochralski silicon (C^3Si) process can be utilized for the growth of crystals with an unusually low oxygen content-typically 1 PPM or less.

2.0 TECHNICAL DISCUSSION

2.1 BACKGROUND INFORMATION

2.1.1 THE NATURE OF OXYGEN IN SILICON CRYSTALS

Currently in the semiconductor industry more than 75% of silicon crystals are grown by the Czochralski (Cz) method. In this method, quartz (SiO_2) crucibles and graphite heaters are used. The surface of the crucibles which are in contact with the silicon melt is gradually dissolved into the melt by the reaction $\text{SiO}_2 + \text{Si} \rightarrow \text{SiO}$. The reaction during crystal growth contributes to the presence of oxygen in silicon crystals.

When a silicon crystal is pulled, oxygen is incorporated into the silicon lattice as a solid solution, with the maximum solubility of oxygen in silicon at its melting point being approximately 2.0×10^{18} atoms/cc. Oxygen occupies the interstitial sites of silicon lattice and gives the bonding configuration of Si-O-Si. However, during heat treatment, oxygen may rearrange within the silicon lattice to form other types of bonding^(1,2). Change in the oxygen bonding leads to changes in the electrical and structural properties of silicon⁽³⁾.

The concentration of oxygen in the silicon crystal is affected by crystal growth parameters. It has been observed that the concentration increases with the rotation of the crystal or crucible⁽⁴⁾. Karimov and Sunyukov⁽⁵⁾ have found that a pull rate of 1.5 to 2.0 mm/min gives the highest oxygen concentration; increasing or decreasing the pull rate from the range leads to a decrease in oxygen content.

Another parameter which effects the oxygen concentration is the pressure of the growth environment. A reduction in furnace pressure tends to decrease the oxygen content in the grown crystal.

The concentration of oxygen in silicon crystals is also affected by the diameter ratio of crystal to crucible. This is manifested by the variation

of oxygen concentration along the axial direction of the tapered section of the crystal. There is strong evidence to suggest that oxygen concentration increases with the ratio of crystal to crucible diameter. Recently, Carlberg et al⁽²⁰⁾ presented a detailed model for the dynamic oxygen level in silicon melts during Czochralski growth which is based on balanced oxygen fluxes through the melt.

The concentration of solid soluble oxygen in silicon can be affected by thermal annealing. This effect becomes obvious when the as-grown crystals are supersaturated with oxygen and the crystals are heat-treated for a prolonged period of time.

There are several harmful affects of the presence of oxygen in silicon crystals. Generation of oxygen donors by heat treatment of 450°C for example, confuses the measurement of dopant concentration via resistivity readings. The presence of oxygen donors will also affect the predicted voltage breakdown and other electrical parameters of a P-N junction device. A second phase precipitate (SiO_2) observed in the heat-treated wafers (700-1000°C) is due to oxygen in the silicon crystals^(3,6). Butler⁽²¹⁾ has described selective heat treating cycles which have proved effective in manipulating and rearranging the interstitial oxygen in the silicon lattice.

Other types of process-induced defects such as swirls and oxidation-induced stacking faults have been correlated to the presence of excess oxygen concentration (< 25 ppm)^(7,8).

In an effort to minimize and hopefully eliminate the problems of oxygen impurity in silicon, various crucible-free methods of crystal growth have been explored with limited success. The Floating Zone method comes closer to eliminating oxygen contamination; however FZ-grown crystals still contain oxygen which is introduced in the feed material, and the problem of oxygen contamination persists.

2.1.2 APPLICATION OF COLD CRUCIBLE/SKULL-MELTING TECHNOLOGY TO THE GROWTH OF SILICON CRYSTALS

The earliest work on cold crucible ("skull") melting was described in a German Patent filed by Siemens and Halske in 1926⁽⁹⁾ and it appears that the technique was not further developed at that time. In 1960, Sterling and Warren⁽¹⁰⁾ reported on their extensive investigations of contamination-free, high temperature melting in several versions of the cold crucible. They showed that by modification of the shape of a water-cooled hearth, the interaction between inducing and induced currents could produce varying degrees of electromagnetic levitation of the melt from the surface of the hearth. The use of cold-boats and crucibles constructed on this principle for zone refining and consolidation have been very successful and they have been well summarized by Bunshah⁽¹¹⁾. Cage crucibles such as that described by Sterling⁽¹⁰⁾, Rummel⁽¹²⁾ and Hukin⁽¹³⁾ have been used successfully for the growth of silicon crystals via the Czochralski technique. It is worth noting however, that these particular cold-crucible configurations are designed to fully-levitate the melt and are, therefore, quite limited in the weight or volume of liquid which can be supported by the RF field.

In 1966, Blicek And Reboux⁽¹⁴⁾ first reported upon a "skull" approach to the melting of silicon. In this case, the silicon melt is confined in a water-cooled, cold-crucible structure and the molten column is radially-squeezed by the RF-field and does not contact the walls of the container. The base of the liquid silicon column is supported on the unmelted silicon feed rod so that the melt is not contaminated by contact with any foreign material. Stable silicon melts were achieved, but subsequent crystal growing experiments were not carried out⁽¹⁵⁾. Other than the patent document, details of this work were not published.

In 1977, Ceres Corporation initiated an intensive program (internally funded) to investigate the adaptation of cold-crucible technology to the fusion and recrystallization of high purity silicon. Utilizing a cold-crucible assembly of proprietary design, Ceres researchers succeeded

in producing and maintaining relatively large (~ 400 gms) stable melts of silicon via direct RF induction heating at low frequencies (250-300 KHz).

Large-grain, polycrystalline silicon ingots pulled from melts confined in the cold crucible were analyzed; the resultant purity levels were found to be at least equal to, or in most instances, better than the semiconductor-grade polysilicon feed stock employed. Based upon the demonstrated potential of the basic cold-crucible/silicon process, Ceres embarked upon the present program in an effort to establish the feasibility of producing oxygen-free single crystals of silicon.

2.2 GOALS OF THE PROGRAM

The goal of the program is to explore the feasibility of utilizing a cold-crucible system for the growth of high purity, oxygen-free single crystals of silicon. The work includes the following tasks:

- I - Investigation of previous research on cold crucible technology and in particular, as it relates to the growth of high-purity silicon crystals.
- II - Study the design and construction of presently available cold-crucible assemblies to determine if modifications and/or redesign is necessary to achieve the overall goals of the program.
- III - Design and/or modify selected cold-crucible configurations(s) to permit continuous or quasi-continuous feeding operation.
- IV - Testing and evaluation of the cold crucible assembly for the growth of high purity, oxygen-free silicon crystals.
- V - Investigate and perform theoretical studies as deemed necessary, of the thermodynamics and heat flow patterns as related to:
 - a.) The placement of the cylindrical silicon charge in the cold crucible.
 - b.) The necessity, configuration and location of a surface thermal reflector.
 - c.) The requirement, design and configuration of seed crystal specimens.
- VI - Delivery of the cold crucible assembly(ies) and silicon crystal specimens.

2.3 RESULTS OF THE PROGRAM

2.3.1 LITERATURE/PATENT SEARCH

An extensive literature/patent search was carried out on RF induction melting and cold crucible technology, particularly as it relates to the production of high purity silicon crystals. Several hundred relevant journal articles and patents have been reviewed, (translated, if necessary), catalogued and indexed.

In addition to specifically related references, a limited number of references were included in fields where technology is relevant to the skull melting process. For this reason, the bibliography includes a number of references on levitation melting, plus a few in the fields of direct induction melting and pedestal-pulling using direct induction heating.

While the resultant bibliography (which is appended to this report) is not meant to incorporate all of the references in the history of the technology, it is hoped that the vast majority of the key references have been included.

2.3.2 DESIGN AND CONSTRUCTION OF THE COLD CRUCIBLE ASSEMBLY

The basic design criteria for the cold crucible assembly was developed during the course of an earlier program supported by the Air Force Cambridge Research Laboratories⁽¹⁶⁾; a theoretical analysis of this system was further developed by Scott and his co-workers Los Alamos Scientific Laboratory⁽¹⁷⁾.

The cold crucible assembly which has been developed during the course of this program is illustrated in Figure 1. It combines the features of the earlier AFCRL system⁽¹⁸⁾ with a bottom-feeding arrangement developed by Ceres Corporation prior to the start of this program for the production of cubic zirconia crystals via the skull melting process.

Basically the cold crucible assembly is a cylindrical structure having an internal diameter of 73 mm and an overall height of approximately 350 mm. Twenty-six 6.35 mm copper tubes (each with a separation of approximately 1/2 mm) form the cylinder. Each closed-end tube is provided with a concentric cooling water outlet; i.e. the cooling water is forced upwards between the walls of the inner and outer tubes and drained via the inner tube. Each of the outer tubes is brazed to a copper, O-ring sealed flange which can be removed to facilitate tube repairs and/or replacement.

The water-cooled base plate of the cold crucible is electrically isolated by a teflon flange from the tubular wall of the structure. The base may be raised or lowered (while the cold crucible is in operation) over a total stroke of 200 mm. Adjustment of base height can be used to vary the height of the silicon melt within the RF coil or alternatively, to introduce poly-crystalline silicon feed stock during the crystal growing operation.

The cold crucible assembly is fastened to the furnace base by the cooling water inlet/outlet tubes which are threaded to accept brass clamping nuts. It is worth noting that the entire cold crucible assembly is electrically isolated from the furnace chamber by O-ring sealed teflon glands and flanges. The cold crucible structure mounted on the furnace base plate is shown in Figure 2.

Selection of an optimum RF coil configuration has been based upon the theoretical analysis presented in Appendix A combined with the experimental data. A closely-coupled 6 turn coil (6.35 mm copper tubing) with an overall height of 65 mm appeared to operate most effectively at the frequency used (250 KHz) for the silicon melting/crystal growing experiments.

The high resistivity of semiconductor-grade polycrystalline silicon feed material at room temperature, does not permit direct coupling by the RF field and some means of preheating must be incorporated into the cold crucible furnace structure. One of the initial methods utilized to preheat

the silicon charge involved the use of a high purity graphite disc positioned above the cold crucible as shown in Figure 3. When the silicon charge is radiatively heated to approximately 800°C to permit direct RF coupling, the graphite disc, which is mounted on a water-cooled stainless steel shaft, can be moved aside (as shown in Figure 4) to facilitate the melting and crystal growing operations. While the graphite disc pre-heater proved to be useful for the melting of the silicon charge, alternative methods of pre-heating were developed which proved to be more advantageous for the subsequent crystal growing operations. The various pre-heater configurations investigated during this program are reviewed in the following sections.

2.3.3 TESTING AND EVALUATION OF THE COLD CRUCIBLE ASSEMBLY

Testing and evaluation of the cold crucible assembly was carried out in the Czochralski-type crystal growing furnace shown in Figure 5. The water-cooled stainless steel furnace chamber is capable of operating at pressures ranging from 10^{-3} torr to 1 atmos.

A manually-operated screwlift mechanism has been incorporated below the furnace chamber to raise or lower the water-cooled baseplate of the cold crucible assembly.

Cold crucible silicon melting experiments which were carried out at Ceres prior to the initiation of the present program indicated that high frequency (3-5 MHz) RF power could not be used to achieve complete melting of the polycrystalline silicon charge confined in the cold crucible. Using a 50 KW output RF power supply operating at approximately 3.5 MHz, a silicon charge (typical weight 450 grams) could not be melted.

Using a 50 KW output, low frequency (250-300 KHz) RF power supply (which incorporates a variable-ratio, low voltage output transformer to minimize sporadic arcing) we were successful in producing and maintaining stable melts of silicon (approx. wgt. 750 gms) which were confined by, but not in physical contact with, the tubular wall of the cold crucible assembly.

During the course of this program silicon melting experiments were also carried out utilizing a 120 KW output, low frequency (250-300 KHz) direct tank-loaded RF power supply which provided a significantly higher output voltage on the work coil. While the coil-to-load spacing was more critical to prevent sporadic arcing, the resultant RF field appeared to be more efficient in terms of coupling and in addition, increased the apparent melt-levitation.

The typical start up and operating procedures used to produce a silicon melt confined in the cold crucible are as follows:

- a.) Discs of semiconductor-grade polycrystalline silicon feed rod (Hemlock Semiconductor Inc.) approximately 20 mm thick (nominal diameter 70-72 mm) which have been cleaned and etched after slicing are stacked on the baseplate of the cold-crucible assembly (total weight approximately 800 gms.).
- b.) The graphite disc preheater is positioned over the charge (as shown in Figure 3) and the furnace chamber is sealed and evacuated.
- c.) Following several flushing and evacuation cycles, the chamber is filled with argon/5% hydrogen to approximately 1 atmosphere.
- d.) The RF power (at 250 KHz) is turned on and coupled to the graphite disc preheater which is heated rapidly to approximately 1000°C. Within a few minutes, the upper surface of the silicon charge is radiantly heated sufficiently to permit direct RF coupling and the preheater is moved aside as shown in Figure 4.
- e.) Silicon melting progresses slowly from the wall of the silicon discs inward. Early stages of melting resemble a mushroom shape i.e. a solid silicon core (or stem) topped with a solid silicon cap surrounded by the melt which is not in contact with the inner surface of the cold crucible.

f.) The thin solid silicon cap is the last remnant of the charge to be melted; a typical melt is shown in Figure 6.

It is worth noting that the silicon melt (approx. 73 mm diam. and 60 to 70 mm deep) is supported on an unmelted disc of the polycrystalline silicon feed material which is in direct contact with the water-cooled cold crucible base plate. However, the RF field exerts a uniform squeezing force on the side of the cylindrical melt with the result that there is a visible separation (approx. 1/2 mm) between the melt and the water-cooled cold crucible.

The silicon melting experiments were terminated by merely shutting off the RF power. Typical quenched silicon melts are shown in Figure 7. The imprint of the copper tube wall structure on the wall of the ingots is the result of the sudden removal of the RF field and the relaxation and freezing of the melt against the copper tube wall of the cold crucible. Spectrographic analyses of recrystallized silicon charges indicate that there is no contamination of the melt by the water-cooled, copper tube structure of the cold crucible.

2.3.4 RESULTS OF SILICON CRYSTAL GROWING EXPERIMENTS

Initial attempts to pull single crystals of silicon by the Czochralski technique from melts confined in the cold crucible resulted in the growth of large-grain polycrystalline ingots as shown in Figure 8. Wetting of the silicon seed crystal by the melt proved to be extremely difficult to achieve. When the etched seed (5 x 5 x 50 mm/(111) and (100) orientation) was immersed into the melt and held for approximately 30 minutes prior to withdrawal, there was no evidence of seed wetting or melting.

Attempts to raise the melt temperature by increasing the applied RF power proved to be fruitless i.e. at the maximum RF power level (50 KW) it was not possible to initiate seed wetting. The furnace system incorporating the cold crucible was then transferred to a 120 KW output, direct tank loaded, RF power supply (operating at 250-300 KHz) and a series

of experiments was initiated in an effort to increase the silicon melt temperature. Despite the application of higher RF power levels (up to 100 KW), the silicon melt temperature was not increased sufficiently to promote seed wetting. However, we did cause intermittent boiling of the cooling water flowing through the cold crucible.

We were forced to conclude that it was not possible to raise the temperature of molten silicon significantly by direct induction heating techniques. Our experimental results were confirmed upon examination of the electrical conductivity data for solid/molten silicon⁽¹⁹⁾ i.e. solid silicon @ 1420°C has an electrical conductivity of 316 mho/cm which increases dramatically to 10^4 mho/cm for molten silicon at the same temperature. The very high electrical conductivity of molten silicon thus effectively limits the low frequency RF power input to the melt.

In light of the practical difficulties encountered in raising the silicon melt temperature by merely increasing the applied RF power, we decided to explore the alternative of independantly heating the seed to promote seed-wetting. A resistance-heated silicon seed holder as shown schematically in Figure 9, was designed and constructed. A miniature nichrome resistance heating element was contained within a transparent quartz tube enclosing the upper end of the silicon seed. Power leads for the resistance-heating element were incorporated in a hollow, stainless steel seed shaft. Ceramic-to-metal gas-tight seals were used to terminate the power leads within the chamber. Since the seed shaft must be raised or lowered and rotated simultaneously, a slip-ring assembly is attached to the exposed end of the seed shaft to transmit power from a Variac control to the heating element.

A series of silicon crystal growing experiments were carried out using the resistance-heated seed holder (see Figure 10). At heater temperatures of 800-900°C, wetting of the seed by the silicon melt was improved marginally but it was apparent that a higher-temperature heat source was necessary to insure reproducible seeding of the silicon melt.

The mechanical and electrical requirements of a larger (higher temperature) resistance-heated seed holder imposed significant practical limitations not only on the furnace system, but on the crystal pulling operation itself, and a decision was made to explore alternative methods of seed heating.

A conical graphite reflector was designed and constructed as shown in Figure 11. Fabricated from high purity (AVC Grade) graphite, the truncated cone incorporates two (2) small viewing ports and rests on three (3) quartz rod supports, and is heated directly by the RF field. Initial tests indicated the need to incorporate a graphite wool insulation on the outer surface of the reflector to reduce the radiant heat loss. The use of the graphite reflector/radiator eliminated the need for the graphite disc preheater as shown in Figure 3.

Utilizing the graphite reflector/radiator, we succeeded in raising and maintaining the silicon seed temperature sufficiently to promote wetting of the seed by the melt and the controlled growth of single crystals of silicon. Two single crystals of silicon grown with the reflector/radiator in place are shown in Figures 12 and 13. The crystals were pulled at a rate of 2.5 cm/hr with a seed rotation rate of 6 RPM. It is worth noting that the resultant crystal diameters were manually controlled. The maximum crystal diameter is physically limited by inner diameter (~ 40 mm) of the top of the graphite cone.

When withdrawn rapidly from the melt, the crystal/melt interfaces appear to be flat and planar (Figure 14). We have also noted that as the melt level is lowered during the crystal pulling operation, the core of the growing crystal remains molten while the outer surface continues to solidify as a single crystal; this effect is shown in Figure 15. When the growing crystal is rapidly withdrawn from the melt, the molten core spills out leaving a cavity up to 2 cm deep. The solid crystal/melt interface is planar and the single crystal wall is approximately 2 mm thick. It appears that as the melt is lowered within the RF coil during the crystal

growing operation, the RF field (@ 250 KHz) remains coupled to the molten core of the crystal while the surface is cooled by radiation and solidifies. It is anticipated that the molten core phenomenon can be eliminated by maintaining a constant melt level throughout the crystal growing operation by actuating the baseplate lift mechanism (i.e. bottom-feeding).

Use of the graphite reflector/radiator assembly has resolved, to a great extent, the problem of maintaining an appropriate seed temperature to permit wetting by the silicon melt. Figure 16 shows a few of the silicon crystals grown during the course of the program using this technique.

While we succeeded in our primary goal of producing Czochralski-grown silicon crystal with a very low oxygen content (ranging from 0.1 to 5 PPM), analysis of the crystals showed a marked increase in carbon contamination (up to 50 ppm in some cases). Since the graphite radiator/reflector assembly (with its graphite wool insulation) is the only source of carbon contamination within the cold crucible furnace system, we decided therefore, to replace the conical graphite assembly with one fabricated from high purity molybdenum. Figure 17 shows the the molybdenum cone in place (surrounded by graphite wool insulation); unfortunately, it appeared that the molybdenum cone (operating at approximately 1600°C) reacted with the vaporized silicon (or the sub-oxide) to form a low melting eutectic thus destroying the molybdenum cone.

2.3.5 CHARACTERIZATION OF SILICON MATERIAL

Representative samples of the polycrystalline silicon feed material (Hemlock Semiconductor Corp.) and crystals of silicon grown from melts confined in the cold crucible were submitted to the Technical Contract Manager (RADC/EMS) for characterization and analysis.

These samples were, in turn, provided to an RADC vendor (Manlabs Inc.) and the Air Force Wright Aeronautical Laboratories (AFWAL/MLPO) for analysis. In addition, with the permission of the Technical Contract Manager, crystal

samples were also submitted for analysis to the Siltec Corp. (Mr. Al Ozias, Manager of the Silicon Technology Center).

While the characterization results proved to be somewhat inconsistent from one analytical group to another, we can offer the following conclusions:

- Silicon crystals grown from melts confined in the cold crucible exhibit very low oxygen contamination. Oxygen content reported on the samples ranged from a high of 18 PPM (Manlabs Inc.) to "none detected" (AFWAL); the average O₂ content found was approximately 1 PPM.
- Typically, the dislocation densities of the crystals were quite high ($3 \times 10^3/\text{cm}^2$ to $4 \times 10^4/\text{cm}^2$). However, one of the samples tested to date (silicon crystal #47) was found to be dislocation free (AFWAL).
- All crystal samples tested showed significant carbon contamination (ranging from 2-30 PPM).
- The silicon melt was not contaminated by the water-cooled, copper cold crucible assembly. Typically, the level of copper found in the grown crystal was less than that contained in the poly-silicon feed material.

The detailed results of the analyses are summarized below:

- MANLABS RESULTS

- Dislocation Densities

A single crystal of silicon (111) orientation, was sectioned and then etched in order to obtain a measure of the dislocation densities in the crystal. The etch pit count of surfaces that have been etched using the dash etch (5:3:3::HNO₃:HF:CH₃:COOH) followed by a Sirtl decoration etch provide densities in the range of 10⁴ to 10⁵ dislocations per square cm.

Chemical Analysis - Trace Elements

Samples in the form of long, needle-like rods were cut from both the starting polycrystalline material and from the single crystal grown from the starting material. The samples were then used as the electrodes in a mass spectrographic instrument and an analysis made of all possible elements in the two types of silicon.

In order to eliminate any surface contaminants, the "electrodes" were etched in Ultec $\text{HNO}_3 \cdot \text{HF} \cdot \text{Dist. H}_2\text{O}$ reagents and rinsed in deionized water and dried with acetone. The sample electrodes were then presparked for about 15 minutes before experimental spectra were recorded. The normal procedure is to record several spectra at different exposure conditions during the sparking of the electrodes such that experimental variables can be calibrated during the analysis. It was found that during such sequential recording during the sparking of these samples, the electrodes showed considerable inhomogeneity along the length of the electrode. The inhomogeneity noted for several types of elements are indicated in the data.

MASS SPECTROGRAPHIC ANALYSIS

(Concentration in parts per million by weight)

<u>Element</u>	<u>Polycrystalline** Silicon</u>	<u>Crystal #45</u>	<u>Crystal #93</u>	<u>Crystal #103</u>
B	0.03	0.01	0.01	0.04
Na	-	*1	< 0.05	< 0.05
Mg	-	*1	0.05	0.5
Al	2.0	*0.3	0.03	0.5
P	0.5	0.3	0.05	0.1
Cl	0.5	0.5	0.03	0.05
K	4.0	1.0	< 0.05	< 0.05
Ca	0.7	1.0	0.04	0.04
Cr	*0.4	*10	< 0.05	< 0.05
Mn	0.02	0.2	< 0.05	< 0.05
Fe	1.0	0.6	0.4	1.0
Ni	0.4	0.4	< 0.05	< 0.05
Cu	*0.4	*1.0	0.05	< 0.03

*These elements were inhomogeneously distributed along the length of the sample electrode. It was also noted that carbon was detected but the data are qualitative and did show similar inhomogeneities.

**Hemlock Semiconductor Corporation, poly-silicon Lot No. SB070189.

Carbon/Oxygen Contamination

The total carbon and total oxygen content in these samples have been determined using a LECO combustion-type apparatus. The data from the measurements are:

	<u>% Carbon</u>	<u>% Oxygen</u>
Polycrystalline Silicon	0.0064 0.0073 0.0079 (0.0072)	0.0021 0.0023 (0.0022)
Crystal #45	0.0153 0.0126 0.0143 (0.0140)	0.0010 0.0025 0.0019 (0.0018)

Triplicate determinations are made when the difference between the first two data points show significant spread in their values. In terms of other units, note that 0.0072%, for example, is the same as 72 ppm.

These data suggest that during the growth process, the silicon seems to have picked up some carbon, with the value increasing from 72 ppm to 140 ppm. There does not seem to be any significant difference in the oxygen levels in the two types of materials. Both materials have oxygen concentrations in the 18 to 22 ppm range.

The determination of the oxygen and carbon in samples of this same material have also been made using a fast Fourier transform infrared transmission method on 2 mm thick sections of unpolished material. The data is as follows:

	<u>Carbon</u>	<u>Oxygen</u>
Polycrystalline Silicon	1.2 ppm	4.9 ppm
Crystal #45	2.6 ppm	5.8 ppm

The data obtained from the IR transmission measurements are based on a series of calculations defined by the position of the transmission maximum in the spectra. Hence, the position of that peak can significantly influence the background corrections, the shape and sharpness of the peak and several other experimental factors. The data presented here were obtained from only one measurement on one sample that may not have been optimized in the surface preparation. The data are very semi-quantitative but can be used to compare the two materials.

The FFT data suggest that the single crystal material does contain more carbon than the starting material. The ratio of the carbon concentrations are about the same for the FFT data and LECO combustion data.

The spectra obtained from these silicon samples did show a relatively strong transmission band near the spectra range of about 1200 wave numbers. The bands in that region are normally associated with silicon oxygen bands

found in many of the silicon oxide-type compounds. Because of that band, due to a precipitated form of the oxide (silicon oxide or dioxide, for example) it is difficult to estimate the concentration of the interstitial oxygen. The values reported for these samples could be in error due to this interference. The data, however, do suggest that the oxygen concentration in the two samples is about the same.

The exact relationship between the two methods of determining the carbon and oxygen content in silicon is a subject still under discussion. Hence, the above data should be considered as preliminary and semi-quantitative.

The combustion method was used to determine the concentration of carbon and oxygen in two (2) later samples of silicon grown from the cold crucible and the data is presented below:

	<u>Carbon</u>	<u>Oxygen</u>
Crystal #93	< 10 PPM	13 PPM
Crystal #103	10 PPM	16 PPM

● AFWAL/MLOP RESULTS

Samples of single crystals of silicon pulled from melts confined in the cold crucible along with a sample of the polycrystalline silicon feed material were submitted to AFWAL/MLOP for characterization.

The results of their analyses are presented below:

Spark Source Mass Spectrometry (by Charles Evans & Associates, San Mateo, CA)

<u>Element</u>	<u>Polycrystalline Silicon (Hemlock)</u>	<u>Single Crystal Silicon #45</u>	<u>Range Factor</u>	<u>Range Rank</u>
B	0.02	0.02	1	2
Mg	1	---	0	1
Al	0.1	0.05	2	3
Si	Major	Major	---	---
P	0.5	0.05	10	6
Cl	0.05	0.2	4	4
K	---	0.5	0	1
Ca	0.5	0.1	5	5
Cr	0.3(inhomogeneous)	0.02	15	7
Mn	---	0.02	0	1
Fe	0.3	0.3	1	2
Cu	0.2(inhomogeneous)	0.03	10	6
As	---	*0.01		

*Not confirmed.

All concentrations in parts per million.

Elements not detected less than 0.05 parts per million atomic,
unless otherwise noted.

Absolute accuracy \times/\div a factor of 3.

Oxygen and Carbon Analyses

	<u>Oxygen</u>	<u>Carbon</u>
Polycrystalline Silicon (Hemlock)	Not Observed	6.5×10^{16} (1.3 PPM)
Single Crystal #45	Not Observed	1.0×10^{17} (2.0 PPM)
Single Crystal #47	5.2×10^{15} (0.1 PPM)	1.1×10^{17} (2.2 PPM)

The infrared absorption measurements were carried out at approximately 8°K using highly-polished 4 mm thick samples. The sample spectra were then referenced to high purity oxygen and carbon-free silicon spectra to obtain absorption coefficients. Appropriate calibration factors (for 8°K) were then used to determine concentrations.

Hall Data

	<u>Resistivity</u>	N_D <u>(Donor Conc.)</u>	N_B <u>(Boron Conc.)</u>
Single Crystal #45	21.9 ohm-cm	$4.8 \times 10^{18} \text{ cm}^{-3}$	$7.6 \times 10^{14} \text{ cm}^{-3}$
Single Crystal #47	NA	$3.4 \times 10^{13} \text{ cm}^{-3}$	$7.5 \times 10^{14} \text{ cm}^{-3}$

Dislocation Density

Single Crystal #45	$4 \times 10^{14} \text{ cm}^{-2}$
Single Crystal #47	No meaningful dislocations observed.

● SILTEC RESULTS

<u>Crystal Sample</u>	<u>Orientation</u>	<u>Type</u>	<u>RHO</u>	<u>Oxygen</u>	<u>Carbon</u>
#113	111	P	27 cm	1.28	3.48
				0.89	3.34
#121	111	P	1.9 cm	0.79	9.60
				1.04	9.84
#126	100*	P	1.7	0.75	11.44
				1.09	11.69

Notes:

*Crystal twinned.

Oxygen and carbon in parts per million.

ASTM-79 as read on Nicolet MX-1.

(We have little prior experience with repeatability of oxygen readings at this level).

Dislocation densities are in excess of $1500/\text{cm}^2$ on all samples. Each crystal sample showed regions of unstructured growth.

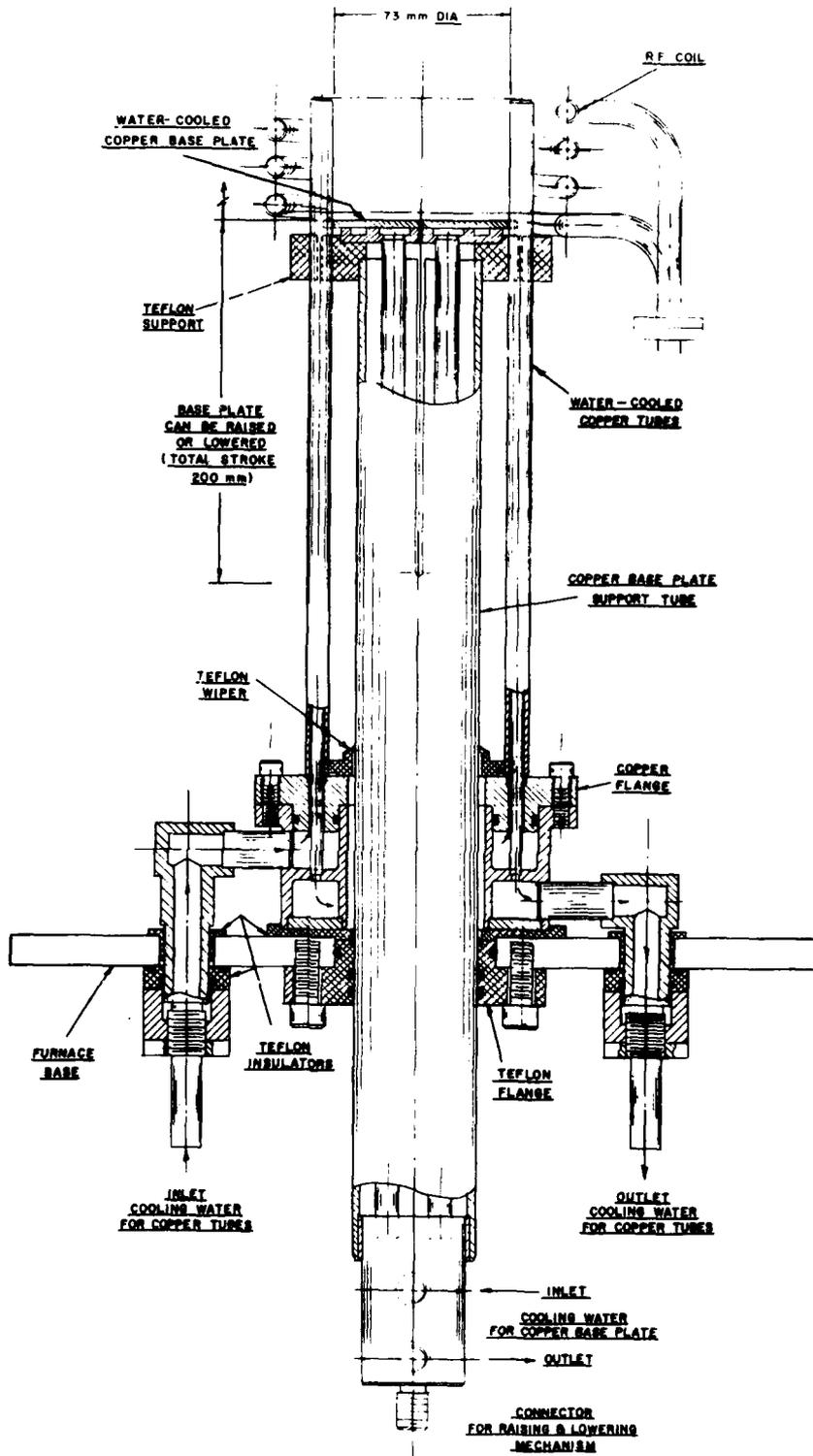


FIGURE 1

WATER-COOLED
COLD CRUCIBLE ASSEMBLY

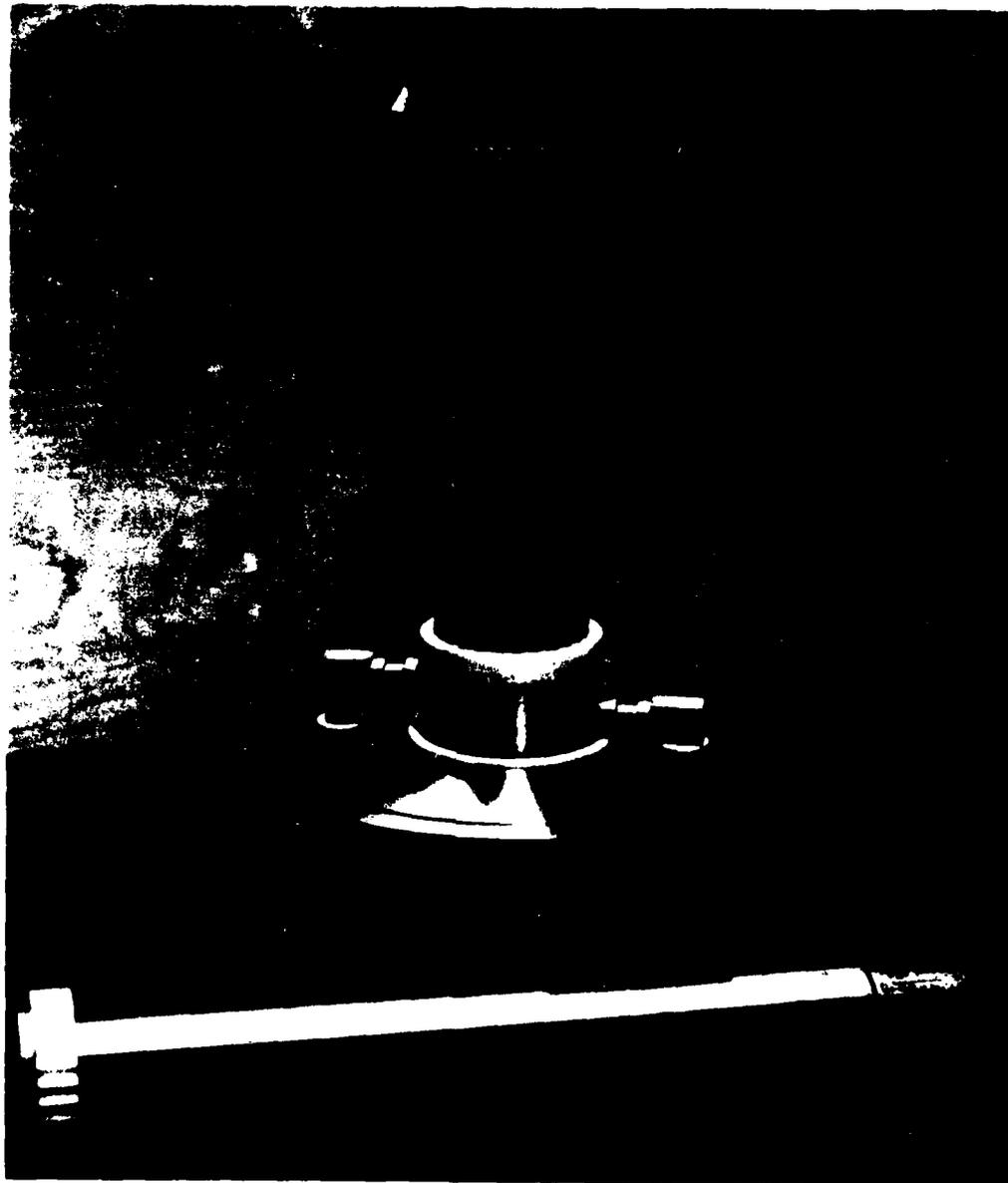


FIG. 1. MECHANICAL ASSEMBLY MOUNTED ON THE FURNACE BASE PLATE

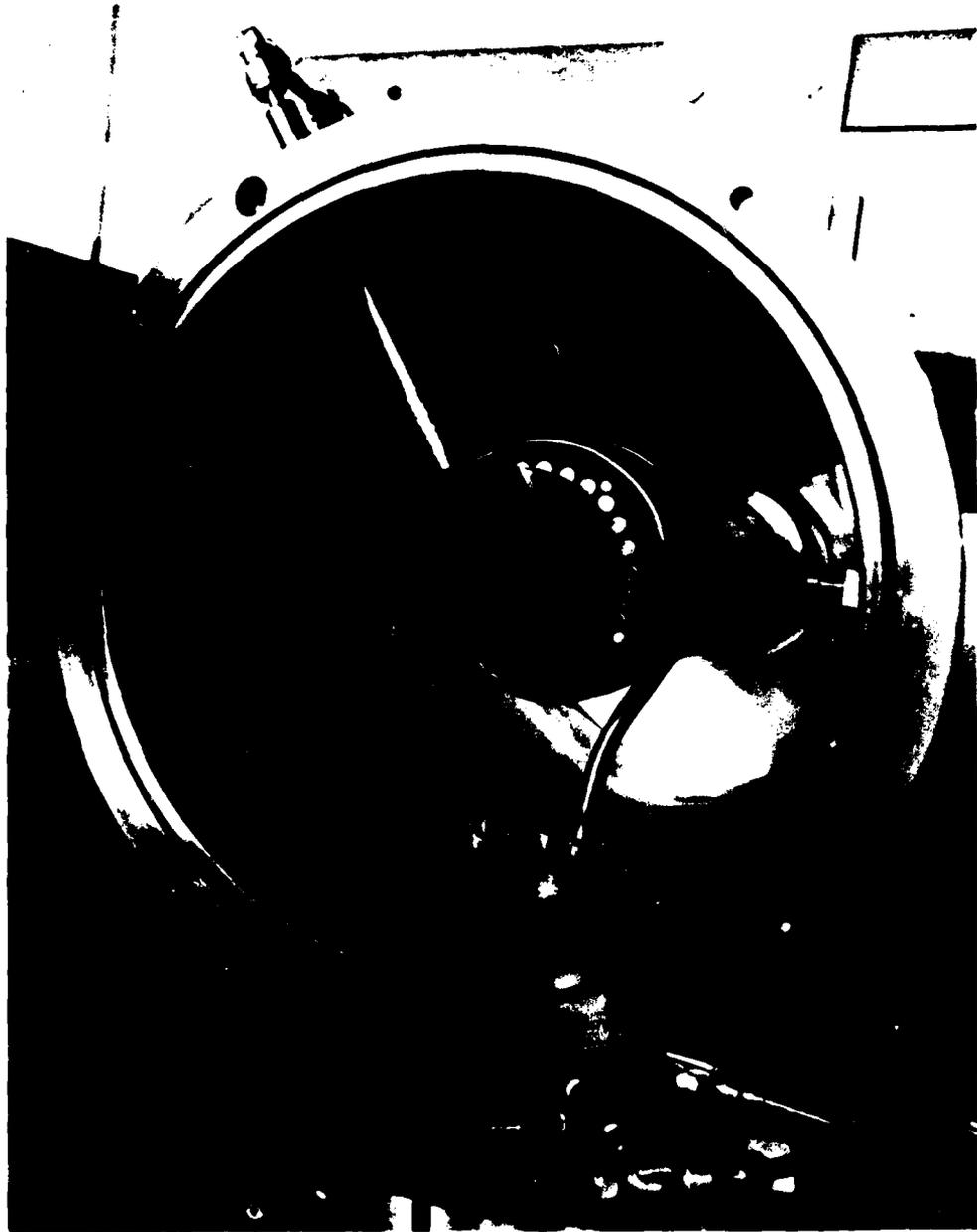


FIGURE 3 GRAPHITE DISC PREHEATER POSITIONED ABOVE THE COLD CRUCIBLE



FIGURE 4 GRAPHITE DISC PREHEATER IN THE DISPLACED POSITION

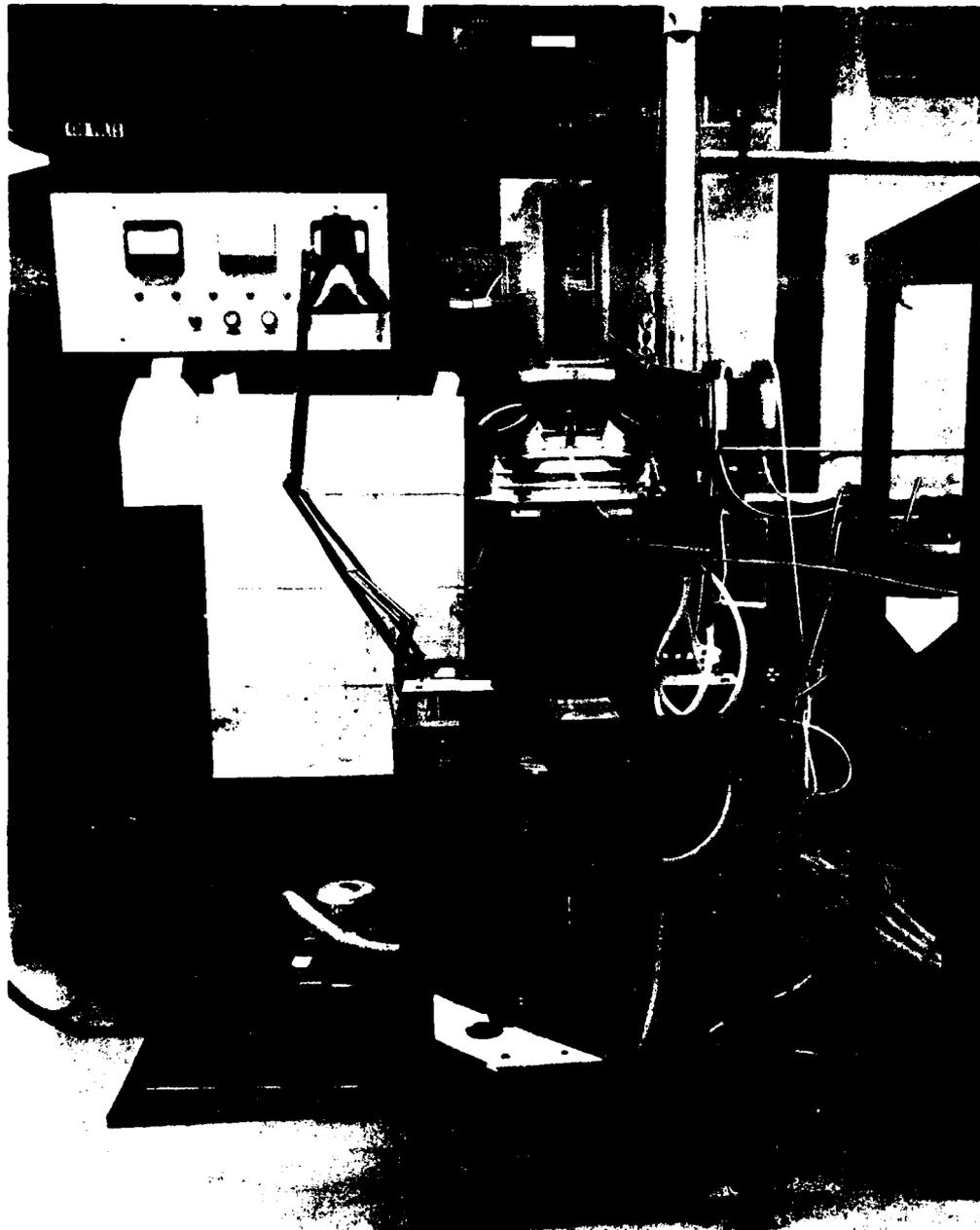


FIGURE 1. VIEW OF FURNACE CONTAINING THE COLD CRUCIBLE ASSEMBLY

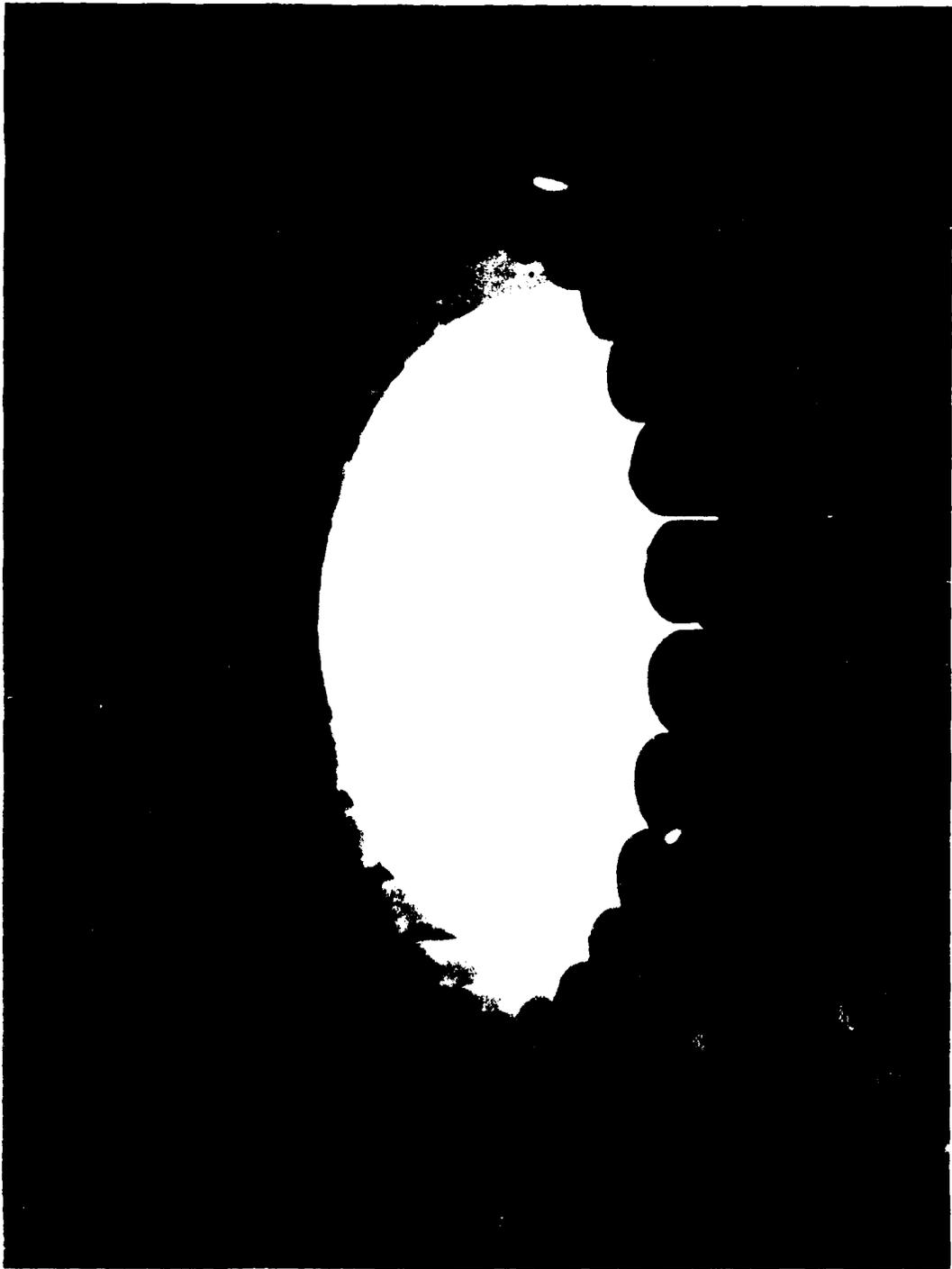


FIGURE 6 SILICON MELT CONFINED IN THE COLD CRUCIBLE

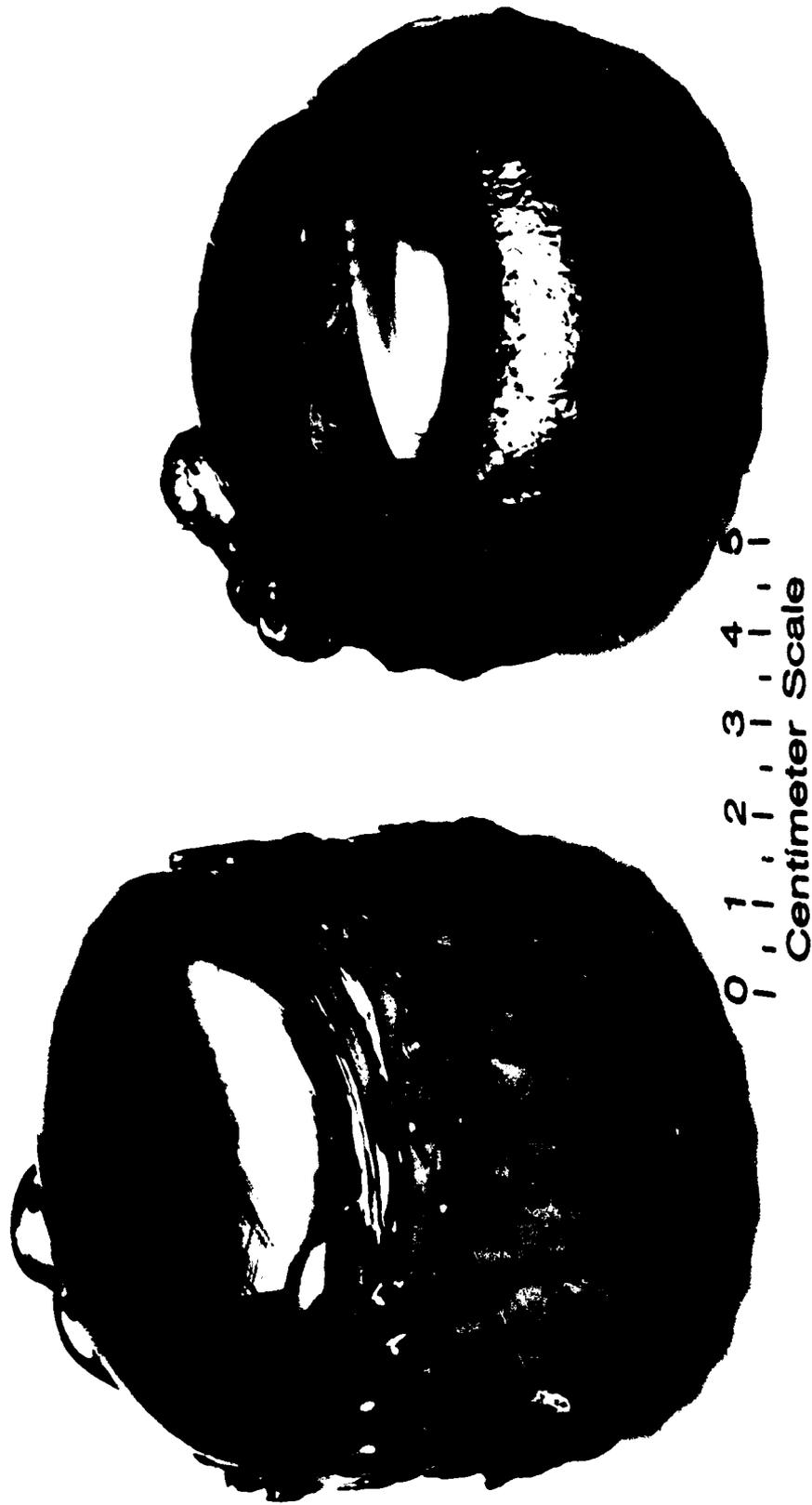


FIGURE 7 QUENCHED SILICON MELTS REMOVED FROM THE COLD CRUCIBLE

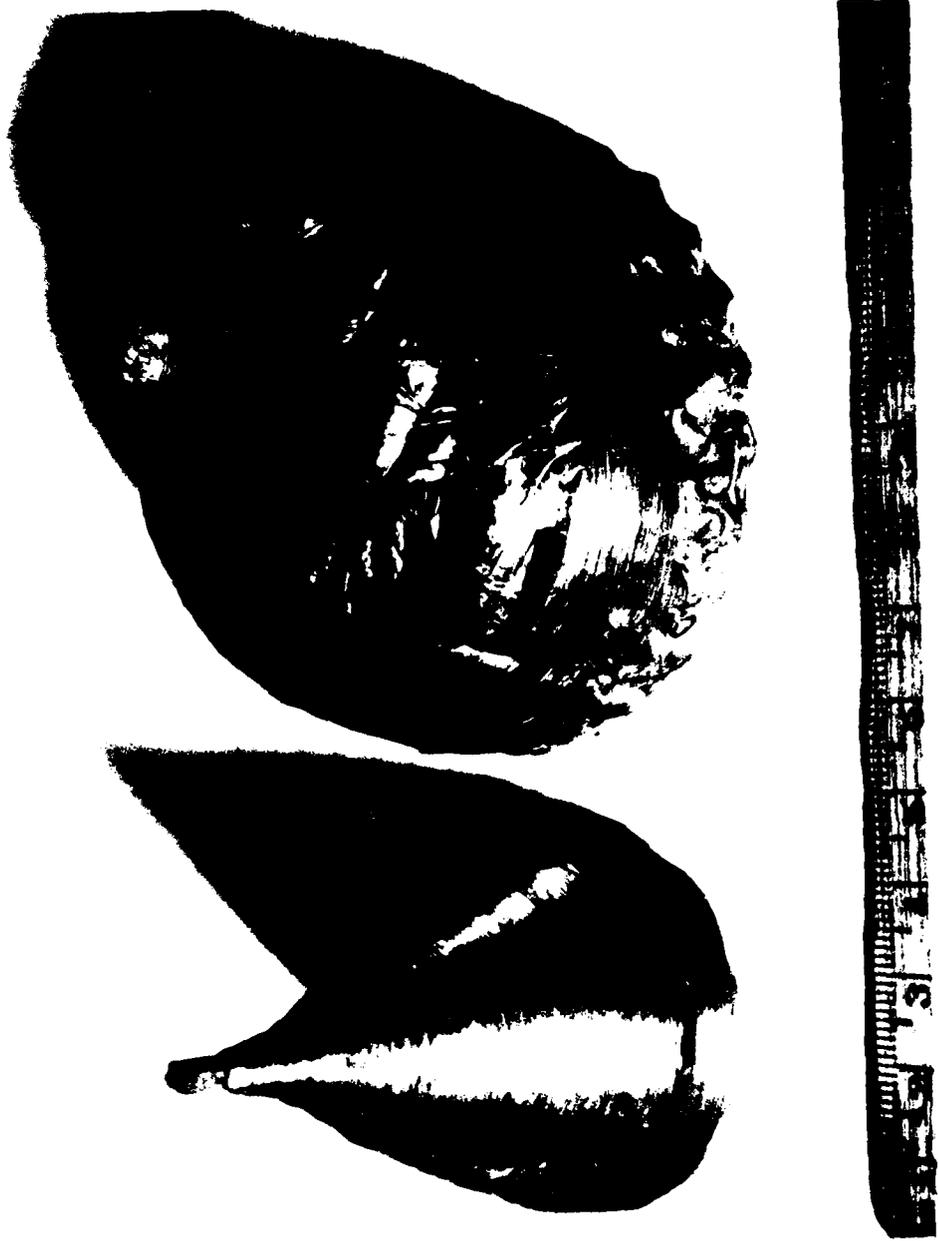


FIGURE 8 POLYCRYSTALLINE SILICON INGOT PULLED FROM THE COLD CRUCIBLE

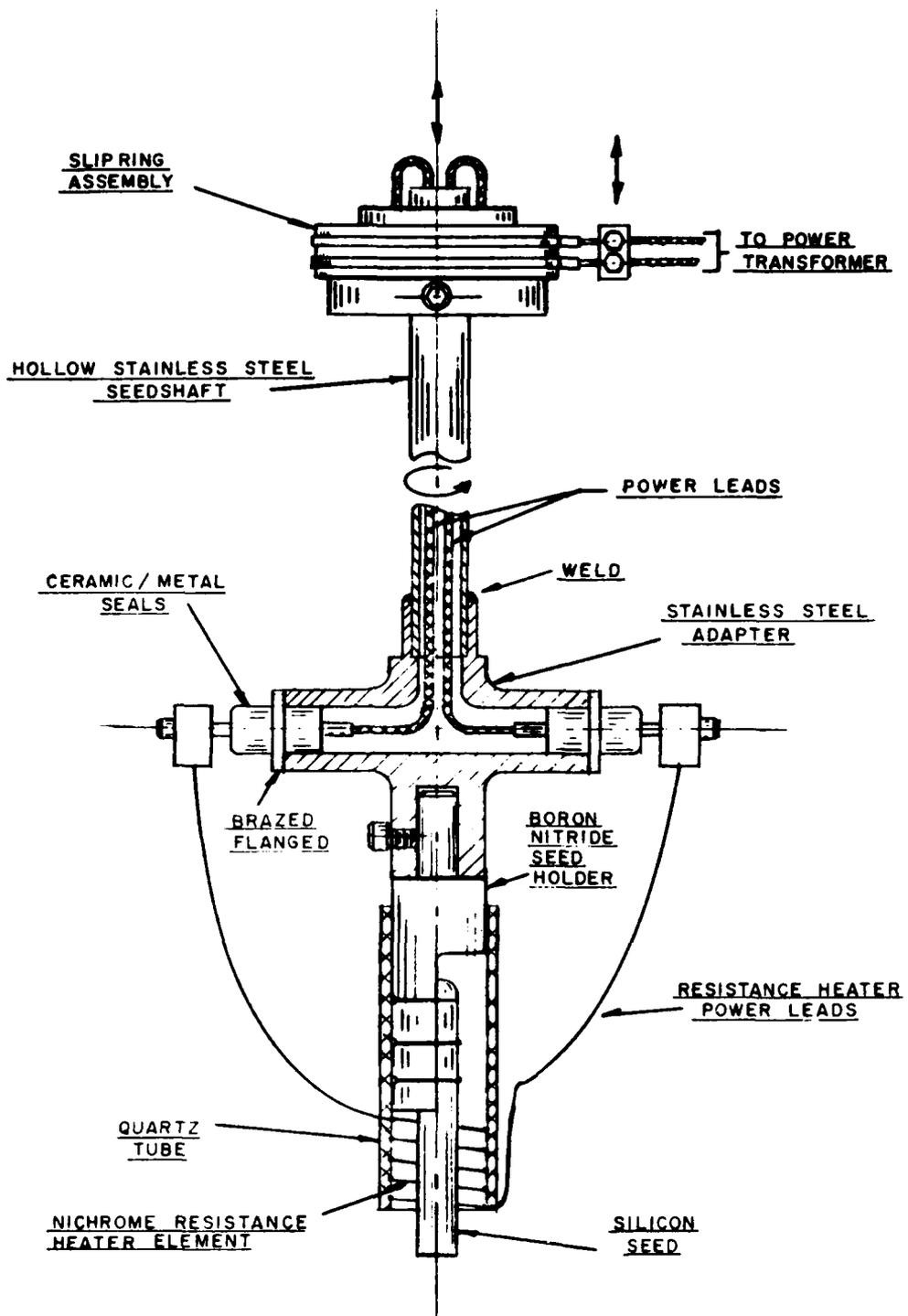


FIGURE 9

RESISTANCE HEATED SEED HOLDER

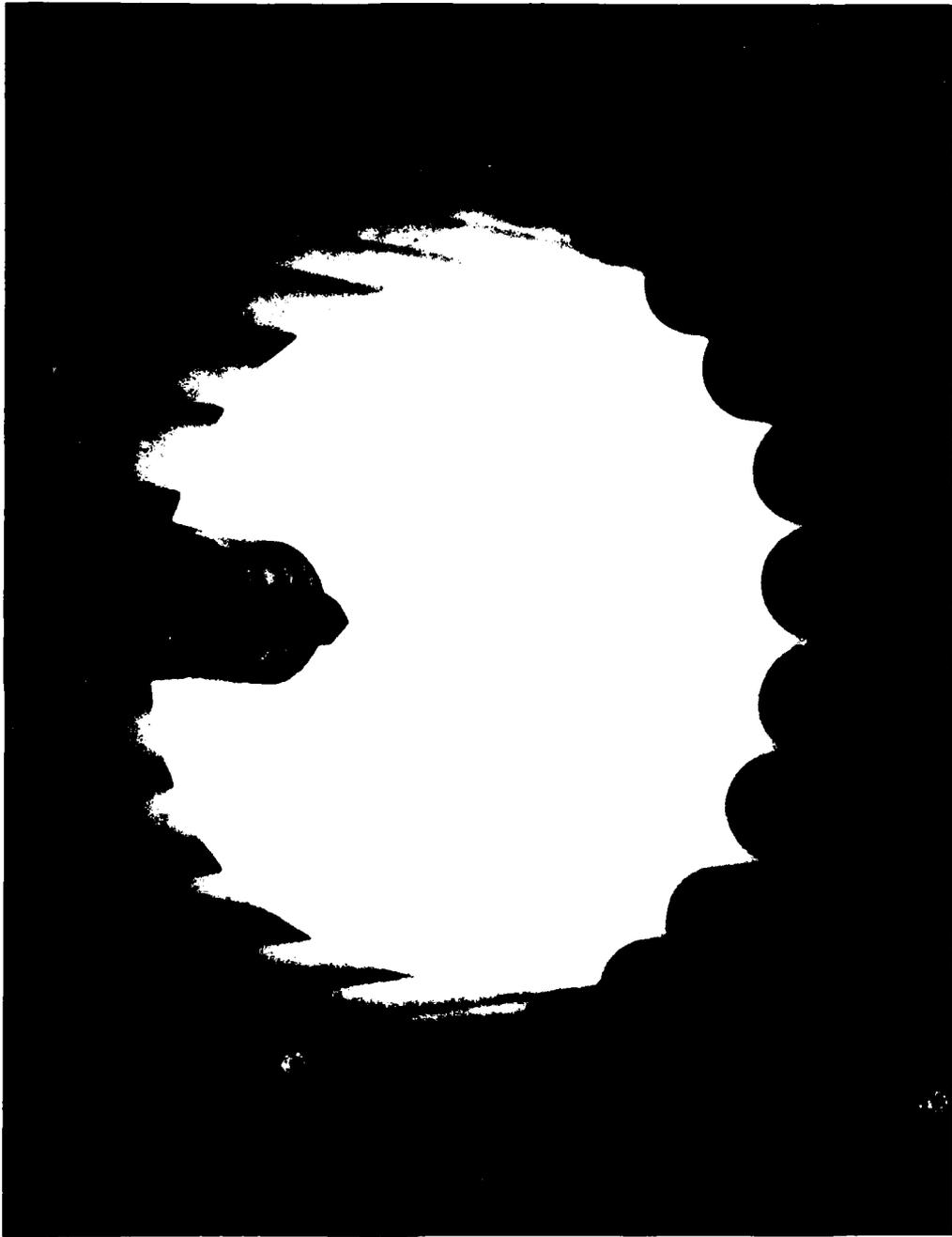


FIGURE 10 CRYSTAL PULLING EXPERIMENT UTILIZING THE RESISTANCE HEATED SEED HOLDER

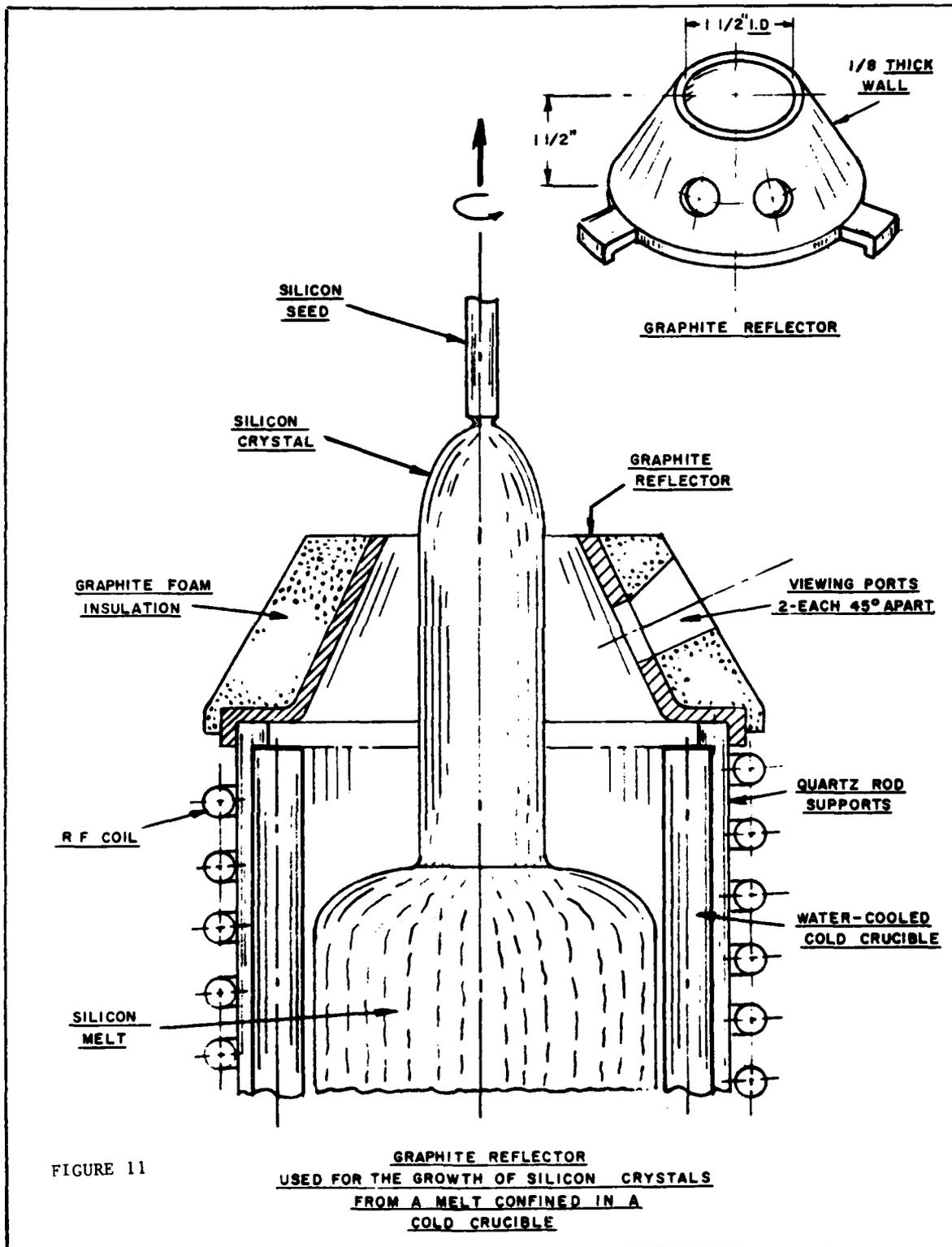


FIGURE 11

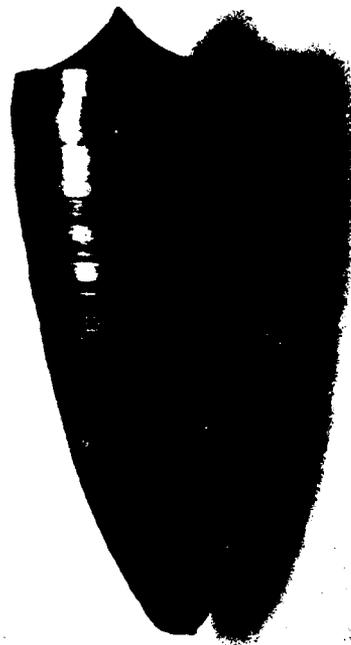


FIGURE 12 SINGLE CRYSTAL OF SILICON GROWN FROM A MELT CONFINED IN THE COLD CRUCIBLE

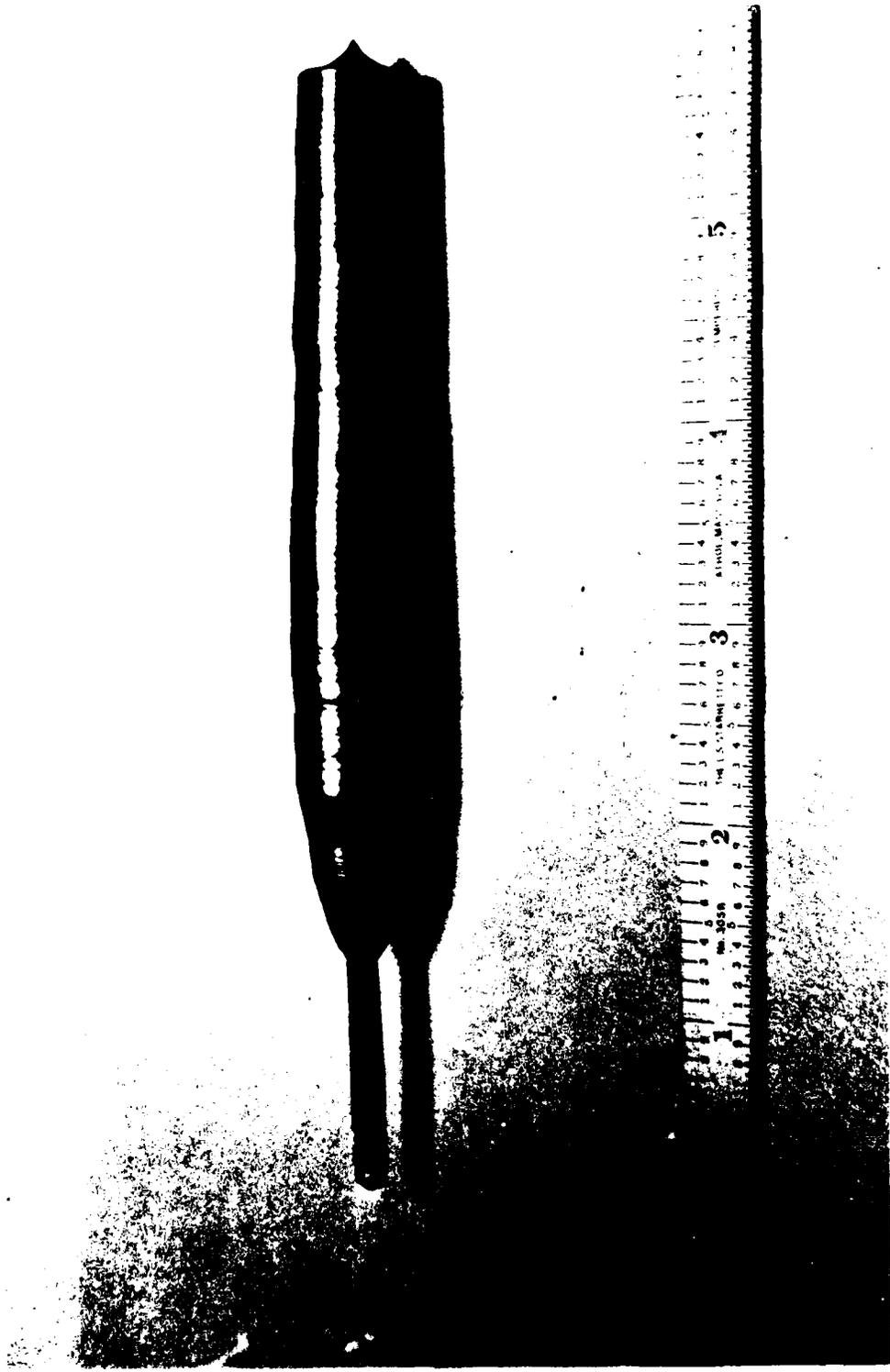


FIGURE 13 SINGLE CRYSTAL OF SILICON GROWN FROM A MELT CONFINED IN THE COLD CRUCIBLE



FIGURE 14 SINGLE CRYSTALS OF SILICON SHOWING THE CRYSTAL MELT INTERFACE

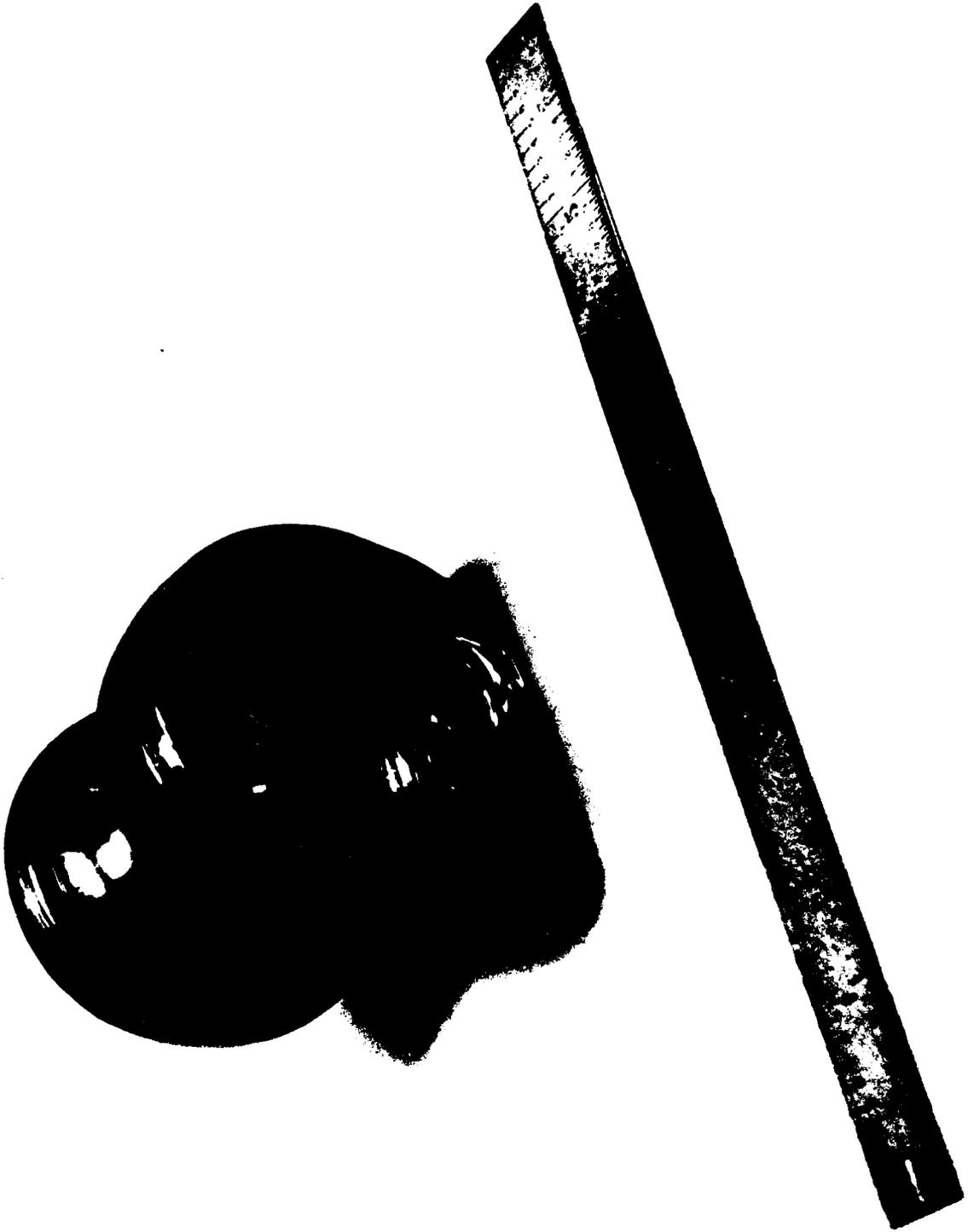


FIGURE 15 SINGLE CRYSTAL OF SILICON WITH HOLLOW-CORE CRYSTAL/MELT INTERFACE

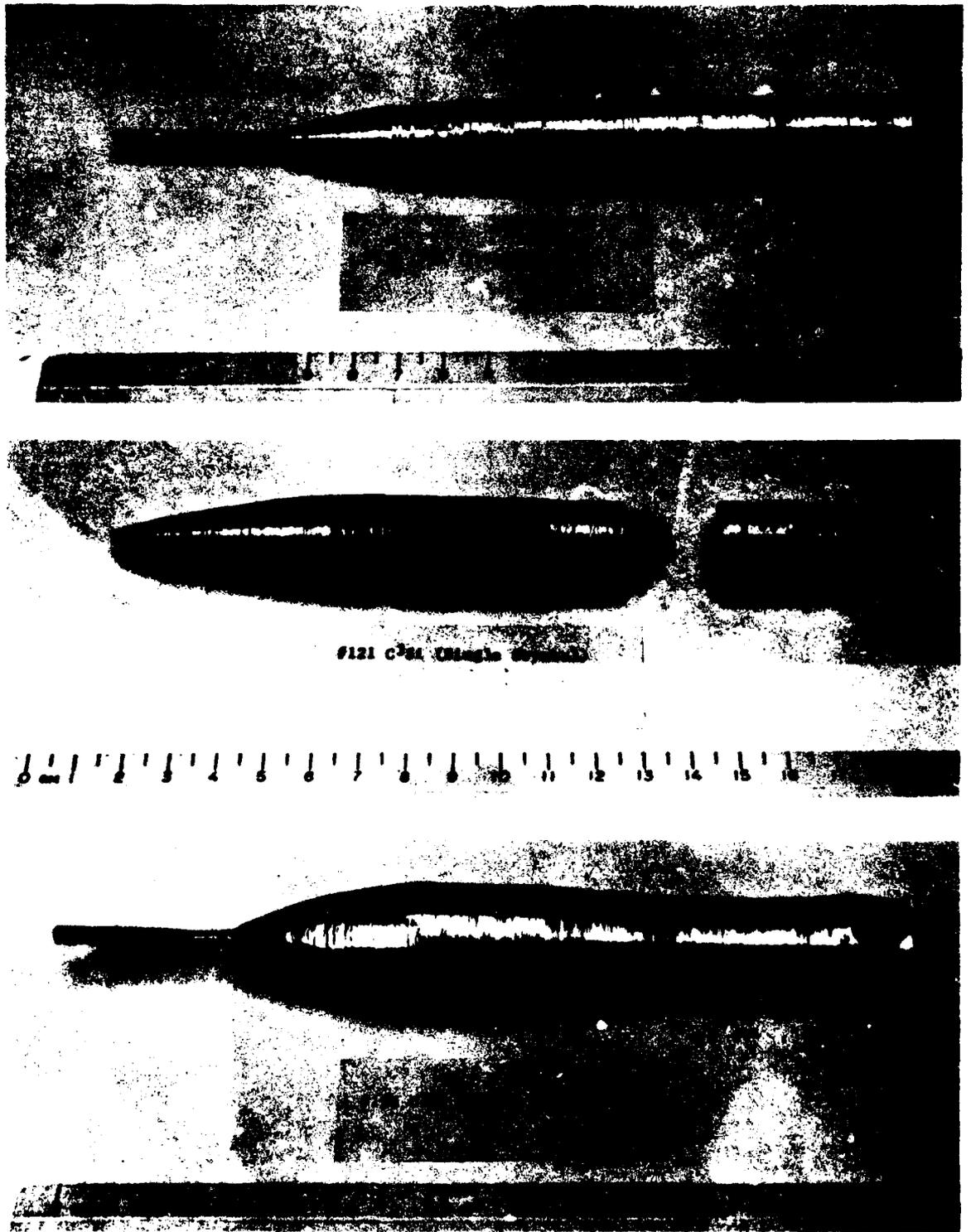


FIGURE 16 CRYSTALS OF SILICON GROWN FROM MELTS CONFINED IN THE COLD CRUCIBLE



FIGURE 17 MOLYBDENUM RADIATOR/REFLECTOR ASSEMBLY

3.0 CONCLUSIONS

- A. The high electrical conductivity of molten silicon (10^4 mho/cm at 1420°C) effectively limits the maximum RF power (@ 250-300 KHz) which can be coupled directly to the melt confined in the cold crucible.
- B. An external heat source is required to raise the temperature of the single crystal seed to insure wetting by the silicon melt. The use of a conical graphite reflector/radiator shield, which is heated by the RF field, has permitted the reproducible growth of single crystals of silicon from melts confined in the cold crucible.
- C. Control of silicon melt level (via bottom-feeding) throughout the crystal growing operation is necessary to prevent the formation of a molten core within the growing crystal.
- D. Analytical results show that single crystals of silicon produced by this technique, have purity levels which are at least equal to, and in most cases, better than the semiconductor grade poly-silicon feed material used. There is no evidence of copper contamination by the water-cooled, copper cold crucible structure.
- E. Single crystals of silicon produced by this technique exhibit very low oxygen contamination. The average oxygen content found was approximately 1 PPM.
- F. The single crystals of silicon produced during the course of this program showed high level of carbon contamination (ranging from 2 to 30 PPM). We believe that the graphite reflector/radiator assembly (which incorporates graphite felt insulation) is the source of this impurity.

G. The typical dislocation density of the crystals produced were relatively high ($3 \times 10^3/\text{cm}^2$ to $4 \times 10^4/\text{cm}^2$). However, we were encouraged to note that at least one of the crystals evaluated (#47) exhibited no detectable defects.

4.0 RECOMMENDATIONS

- A. An external heat source was found to be necessary to raise the silicon seed temperature to insure wetting by the silicon melt. The graphite radiator/reflector assembly is heated by the RF field which also maintains the silicon melt, i.e. it was not possible to control the radiator/reflector temperature independently of the the melt temperature.

Development of a temperature controlled radiator/reflector assembly which is not dependent upon the RF energy applied to the silicon melt, would significantly enhance the control of the seeding and crystal growing operations.

- B. The conical graphite radiator/reflector assembly (with its graphite felt insulation) appears to be the source of the carbon contamination found in all of the crystals which were analyzed. It will be necessary to investigate various grades of graphite (or possibly other materials) used to fabricate this assembly with the goal of eliminating the carbon contamination.
- C. While the crystals grown during the course of the program exhibit unusually low oxygen content, the use of improved furnace evacuation procedures and gas purification systems should be investigated in an effort to further reduce the residual oxygen contamination.
- D. Little attention has been focussed on the growth of low dislocation silicon crystals from melts confined in the cold crucible. With the development of a controllable seed-heating source, it would be appropriate to explore various seed-necking/tapering methods in an effort to produce dislocation-free crystals consistently.

E. The bottom-feeding mechanism which was incorporated in the cold crucible assembly, was adjusted periodically in an effort to maintain a constant silicon melt level. Some means of automatic melt-level adjustment should be incorporated into the system.

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APPENDIX A

WORK-COIL - LOAD COUPLING IN RADIO FREQUENCY INDUCTION HEATING

1. Introduction

The following analysis treats the problem of inductively coupling power into a long cylindrical load in the two extreme cases, i.e. when the penetration depth is large so that the applied radio frequency magnetic field penetrates the load unattenuated, and when the penetration depth is small so that heating is caused by a screening current flowing in a surface layer in the load and there is no magnetic field in the interior of the load. The heating in the first case is inversely proportional to resistivity and so, in silicon, increases with increasing temperature. In the second case heating is proportional to the square root of resistivity and so, in silicon, decreases slowly with increasing temperature.

The problems associated with the intermediate region, the changes in the distribution of heating with changes in resistivity, the resultant temperature distribution, and instabilities which may be caused by these phenomena are not treated here, but are discussed in some of the referenced literature.

In practice, the engineer trying to optimize induction heating in a particular application has little to work with other than the number of turns in the work coil and the choice of whether or not to use a transformer. The output frequency of most induction heaters is tuneable over only a small range and has little effect on heating in this range. When the resistivity of the material in the load is very dependent on temperature, it may be difficult to design a coil which gives good coupling to the load at both

extremes of resistivity. Often the most practical solution is to use an induction heater with a much greater power rating, by a factor of two to perhaps as much as five, than the power needed to heat the load to temperature.

The nomenclature used in this analysis is:

P = power (watts)

V = volume (meters³)

ω = radian frequency = $2\pi f$

μ_0 = permeability of space = $4\pi \times 10^{-7}$ henry/meter

H = rms magnetic field (amperes/meter)

r = radius of the load (meters)

r_c = radius of the work coil (meters)

ρ = resistivity of the load material (ohm-meters)

ℓ = length of the load or work coil (meters)

n = number of turns in the work coil

i = coil current (amperes)

e = induction heater output voltage (volts)

L = loaded inductance of work coil (henrys)

L_T = internal tank coil inductance (henrys)

R_{eq} = equivalent resistance of load (ohms)

N = r.f. transformer turns ratio

L_m = r.f. transformer mutual inductance (henrys)

L_ℓ = r.f. transformer leakage inductance (henrys)

L_{eq} = equivalent output inductance of the r.f. transformer (henrys)

d = penetration depth = $\sqrt{2\rho/\omega\mu_0}$ meters

2. Large Penetration Depths

When the resistivity is high and the frequency is low and, consequently, the penetration depth is large, we may assume that the magnetic field produced by the work coil is not reduced appreciably by screening currents induced in the load. In this case the incremental power per unit volume

induced in a cylindrical load is:

$$\partial P / \partial V = \omega^2 \mu_0^2 H^2 r^2 / 4\rho \quad (\text{watts/m}^3)$$

and the power per unit length induced in a cylindrical load is:

$$P/l = \pi \omega^2 \mu_0^2 H^2 r^4 / 8\rho \quad (\text{watts/m})$$

The heating is concentrated at the surface and is inversely proportional to resistivity. The apparent dependence on the square of frequency will be shown to be untrue.

The design of the work coil in this case is simple if the work coil is also the tank coil. The coil inductance (for long coils) is:

$$L = \mu_0 \pi r_c^2 n^2 / l \quad (\text{henrys})$$

and must fall in a range suitable for use in the tank circuit of the induction heater. The practical range is about an order of magnitude.

The coil current is determined by the coil impedance, ωL . The field, H , is the ampere-turn product per unit length of the coil. Thus:

$$H = ni/l = \frac{ne}{l\omega L} = e / (n\omega\mu_0 \pi r_c^2) \quad (\text{amps/m})$$

and
$$P/l = \frac{1}{8\pi\rho} \left(\frac{r}{r_c} \right)^4 \left(\frac{e}{n} \right) \quad (\text{watts/m})$$

The power is limited by the volts per turn, an induction heater capacity or arc-over limit. Increasing frequency without increasing voltage, decreases the coil current and the magnetic field proportionally so that there is no increase in power. The practical minimum number of turns results in the maximum power into the load. Tight coupling, r_c only slightly larger than r , is desirable since the ratio, r/r_c , is raised to the fourth power in the last equation.

When the inductance heater has an internal tank coil in series with the work coil, the sum of the two inductances determines the coil current and the magnetic field.

$$H = \frac{ne}{\ell_w (L + L_T)} \quad (\text{amperes/meter})$$

An equivalent circuit for the tank coil, work coil, and load is shown in Figure 1. R_{eq} is the equivalent load resistance in parallel with the work coil. Maximum power is now obtained when the number of turns in the work coil makes the work coil inductance equal the tank coil inductance.

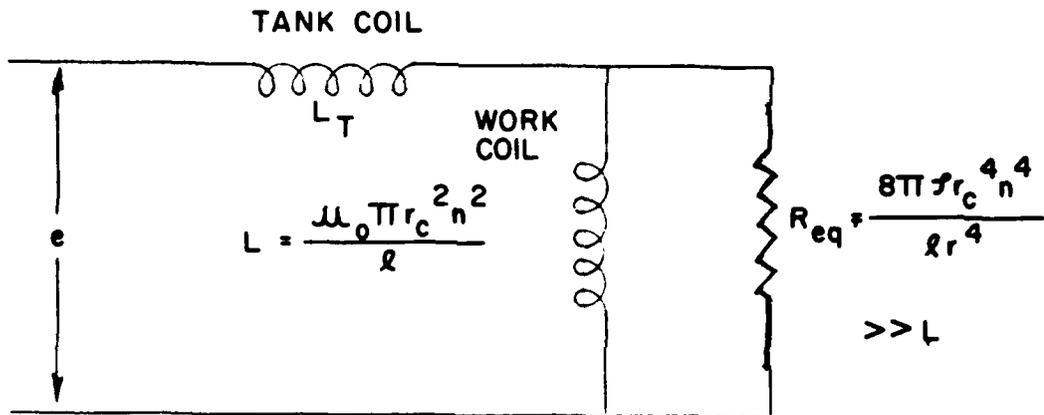


FIGURE - 1
EQUIVALENT OUTPUT CIRCUIT
FOR LARGE PENETRATION DEPTHS

3. Small Penetration Depths

When the resistivity is low and the frequency is high and, consequently, the penetration depth is small, we may assume that a screening current, sufficient to keep the magnetic field out of the interior of the load, flows in a surface layer of the load equal to one penetration depth in thickness. The power per unit length is:

$$P/\ell = \pi r H^2 \sqrt{2\omega\mu_0\rho} \quad (\text{watts/meter})$$

The heating is concentrated at the surface to a greater degree than before and is now proportional to the square root of resistivity. If the work coil is also the tank coil, the current, and consequently the magnetic field, is determined by the impedance of the coil and load together. This impedance consists of the equivalent load resistance in series with the inductance of the loaded coil. Because there is no penetration of the field, the inductance is proportional to the cross sectional area between the coil and the load.

$$R_{eq} = \pi r n^2 \sqrt{2\omega\mu_0\rho/\ell} \quad (\text{ohms})$$

$$L = \mu_0\pi (r_c^2 - r^2) n^2/\ell \quad (\text{henrys})$$

$$i = e/(R_{eq} + j\omega L) \quad (\text{amperes})$$

and

$$P = i^2 R_{eq} = \frac{e^2 R_{eq}}{(R_{eq} + j\omega L)^2} \quad (\text{watts})$$

Some of the parameters still have the same effect. The practical power limit will be the volts per turn. Tight coupling is still important. However, increasing frequency now decreases power by decreasing the field faster than it increases equivalent load resistance. The number of turns in the work coil is not important because it affects L and R_{eq} to the same degree.

If there is an internal tank coil, maximum power is obtained when R_{eq} is made equal to $\omega (L + L_T)$ by using a work coil with the optimum number of turns. An equivalent circuit for this case is shown in Figure 2.

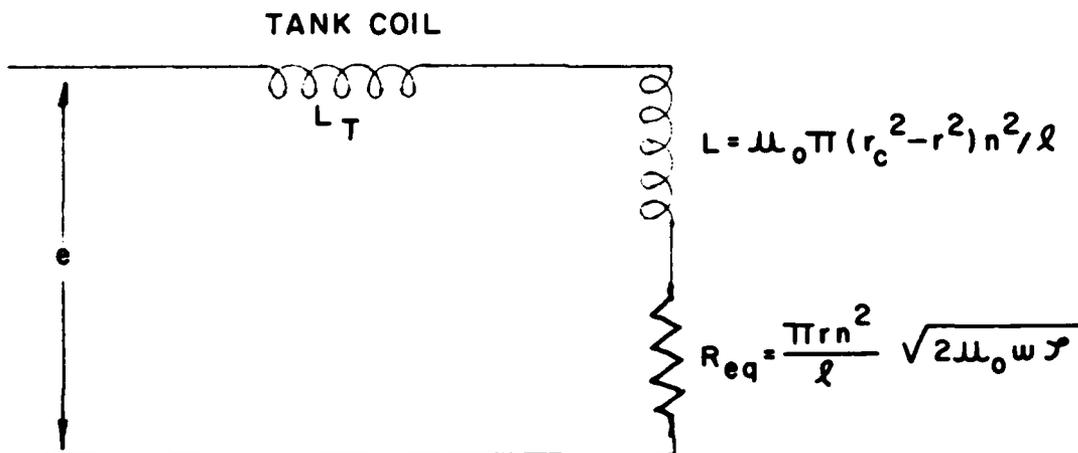


FIGURE — 2
EQUIVALENT OUTPUT CIRCUIT
FOR SMALL PENETRATION DEPTHS

4. Use of Radio Frequency Transformers

In some applications it is not practical to adjust the number of turns in the work coil to make L equal L_T in the case of large penetration depth, or to make R_{eq} equal the combined impedance of L plus L_T in the case of small penetration depth. In these cases a radio frequency transformer can sometimes be used to improve the coupling. Equivalent circuits for the induction heater output circuit with a r.f. transformer are shown in figure 3.

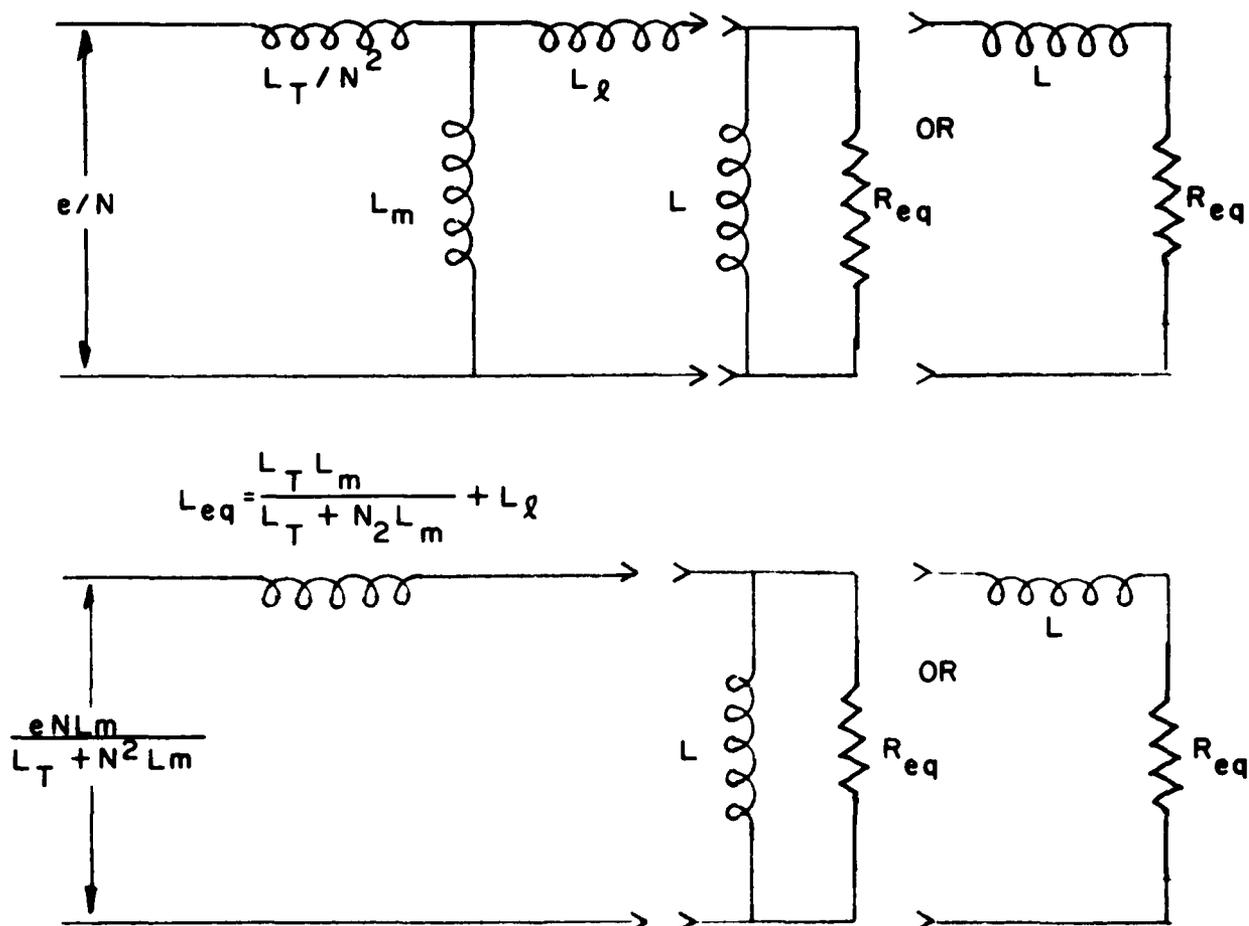


FIGURE - 3
EQUIVALENT OUTPUT CIRCUITS
WITH A RADIO FREQUENCY TRANSFORMER

The choice of r.f. transformers is limited. Turns ratios, N , are about 8:1 to 12:1 and the transformer mutual inductance, L_m is often smaller than one would like. This is because the transformer is often designed to drive a transmission line rather than to optimize coupling.

When a transformer is used, the optimum coil design makes L equal to L_{eq} or R_{eq} equal to ω times L plus L_{eq} in the case of large or small penetration depths respectively. Calculation or experiment must be used to determine whether the transformer with an optimum coil results in better heating than when no transformer is used.

5. Numerical Examples

Consider, as a typical example, the induction heating and melting of a silicon cylinder .035 meter (1-3/8") in radius and .08 meter (3-1/8") long. The induction heater operates at 240 kilohertz, ($\omega = 1.5 \times 10^6$), has a five microhenry internal tank coil and a maximum output of 7000 volts rms (10 kilovolt peak). The resistivity and penetration depth for silicon at various temperatures below and above the melting point are tabulated below.

<u>Temperature</u> <u>°C</u>	<u>ρ</u> <u>ohm-meters</u>	<u>d</u> <u>meters</u>
20	.4	.65
300	.1	.33
1400 (solid)	10^{-4}	.010
1450 (liquid)	10^{-6}	.0010

At the lower temperatures the penetration depth is much greater than the radius and the large penetration depth model is valid. The optimum coil at start up may be found by equating coil inductance to the internal inductance* assuming a 2 inch radius coil is needed for clearance.

$$L = \frac{n^2 r_c^2}{10l + 9r_c} = \frac{n^2 2^2}{31.25 + 18} = 5 \text{ microhenrys}$$

$$n = \sqrt{61.6} \quad \sim 8 \text{ turns}$$

Eight is too many turns for a .08 meter coil length. Tabulated below are the results of using eight and fewer turns in the work coil.

<u>n</u> <u>turns</u>	<u>L</u> <u>uh</u>	<u>H</u> <u>amp/m</u>	<u>P @ 20°C</u> <u>watts</u>	<u>P @ 300°C</u> <u>watts</u>
4	1.3	3.7×10^4	570	2300
6	2.9	4.4	810	3200
8	5.2	4.6	890	3500

The difference in performance between 6 and 8 turns is small and so a 6 turn coil could be used effectively. Note the substantial increase in power with the increase in temperature.

At the melting point, the penetration depth is less than the radius of the silicon and we will use the second model. The equivalent load resistance is:

$$R_{eq} = \frac{\pi r n^2}{l} \sqrt{2\mu_o \omega \rho} = .027 n^2 \quad (\text{ohms})$$

The impedance of the loaded coil is:

$$X_L = \omega \frac{(r_c^2 - r^2) n^2}{10l + 9 r_c} = .064 n^2 \quad (\text{ohms})$$

and the impedance of the tank coil is 7.5 ohms. There is no optimum coil but there is excellent heating anyway. Tabulated below are the equivalent resistance, the inductive impedance and the power into the silicon at 7000 volts.

*A more accurate formula which makes an allowance for end effect and gives inductance in microhenrys for dimensions in inches is used here.

<u>n</u> <u>turns</u>	<u>Req.</u> <u>ohms</u>	<u>$\omega (L + L_T)$</u> <u>ohms</u>	<u>P</u> <u>kilowatts*</u>
4	.43	8.5	290
6	.96	9.8	490
8	1.71	11.6	620

When the silicon melts, the resistivity drops and, consequently, the power into the silicon is also lower. Tabulated below are values for melted silicon, ρ equals 10^{-6} ohm-meter.

<u>turns</u>	<u>Req.</u> <u>ohms</u>	<u>$\omega (L + L_T)$</u> <u>ohms</u>	<u>P</u> <u>kilowatts</u>
4	.043	8.5	29
6	.096	9.8	49
8	.141	11.6	68

From the above numerical examples we may conclude:

- During the start up, when the silicon is near room temperature, heating is poor but improves rapidly.
- During start up, it is important to use a work coil with close to the optimum number of turns, but small deviations from optimum do not seriously degrade coupling into the load.
- Coupling to the silicon is maximum in the intermediate region (where penetration depth is approximately equal to the radius of the load). The models used in this analysis exaggerate the heating in this region.

*These power levels are too high because the penetration depth is not small compared to the radius. The equivalent resistance will be lower than computed and the loaded inductances will be higher than computed. It may be found, in practice, that the induction heater cannot be operated at full voltage under these conditions without overloading.

- Heating becomes poorer as the penetration depth continues to decrease with decreasing resistivity as the temperature of the silicon rises.
- The sudden decrease in resistivity associated with the melting point makes it difficult to heat beyond this point.
- In high temperature, low resistivity region, increasing the number of turns in the work coil will increase heating. There is no theoretical optimum number of turns.
- At some point as resistivity decreases, an r.f. transformer may improve coupling through better impedance matching. A transformer with high mutual inductance is needed to avoid coupling losses in the transformer.

APPENDIX B

RADIATION FROM AN ISOTHERMAL BLACK SURFACE

Dr. Alfred Emslie, Consultant

The following analysis was carried out to determine the nature of thermal radiation losses from the surface of the silicon melt confined in the cold crucible and to determine the influence of the graphite radiator/reflector assembly on the temperature of the melt surface.

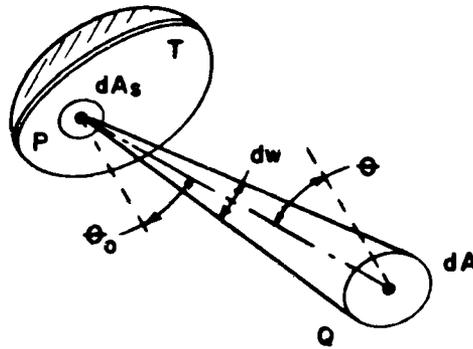


FIGURE -1

Figure 1 shows a black surface ($E=1$), in the form of a cap of arbitrary shape, maintained at a uniform temperature T (deg K). The total power emitted by an element dA_s of the inner surface of the cap, located at the point P , is $\sigma T^4 dA_s$, where σ is the Stefan-Boltzmann constant.

Since the emitted radiation has a cosine distribution about the normal to the element dA_s , the radiation power dW contained in an element of solid angle $d\omega$ at an angle θ_0 to the normal to the surface is given by the formula:

$$dW = \frac{1}{\pi} \sigma T^4 dA_s \cos \theta_0 d\omega \quad (1)$$

where $(1/\pi)$ is a normalized factor.

An element of area dA located at a point Q , at a distance R from P and with its normal inclined at an angle θ to the line PQ , subtends a solid angle:

$$d\omega = \frac{dA \cos \theta}{R^2} \quad (2)$$

at P .

Therefore, from (1) and (2), the power falling on the area dA at Q is:

$$dW = \frac{\sigma T^4 dA_s dA \cos \theta_0 \cos \theta}{R^2} \quad (3)$$

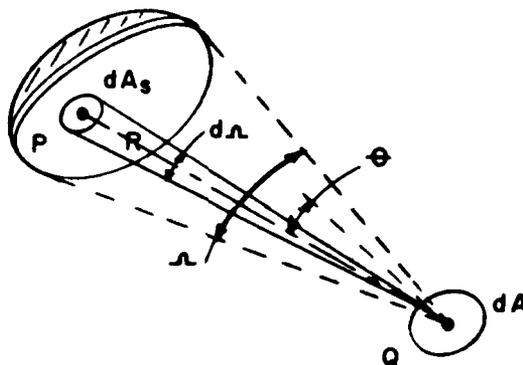


FIGURE - 2

Figure 2 shows that:

$$\frac{dA_s \cos \theta_0}{R^2} = d\Omega \quad (4)$$

where $d\Omega$ is the solid angle subtended by dA_s at Q . Thus Eq (3) becomes:

$$dW = \frac{\sigma T^4 dA \cos \theta d\Omega}{\pi} \quad (5)$$

Therefore the total power received by dA is:

$$W = \sigma T^4 dA \int_{\Omega} \frac{\cos \theta}{\pi} d\Omega \quad (6)$$

Since θ_0 and dA s do not appear in this formula, the power W depends only on the boundary of the radiating cap and not on the shape of the surface of the cap, provided that the field of view of the point Q does not include any part of the outer surface of the cap. Exactly the same argument applies for a cap containing a hole, as in the case of the truncated graphite cone. The integral in Eq (6) depends only on the two boundaries and on the shape of the surface connecting these boundaries, again provided that only the inner surface of the cap is seen from the point Q .

RADIATION FROM THE GRAPHITE CONE

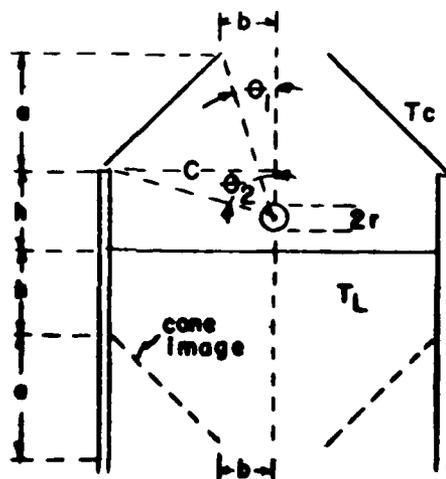


FIGURE - 3

Figure 3 shows the graphite cone, of height a , and radii b and c , placed on top of the cold crucible tubes and maintained at an absolute temperature T_c . The surface of the liquid silicon is located at a distance h below the bottom of the cone and has a uniform temperature T_L .

In order to find the effect of the cone we calculate 1) the total radiant power falling on a small black sphere of radius r located just above the center of the liquid surface, and 2) the equilibrium temperature of the sphere.

The power reaching the sphere consists of three contributions: 1) the direct power from the cone, 2) the direct power from the liquid surface, and 3) the power from the cone that is reflected by the liquid surface. Radiation contributions from the cold crucible and from the aperture at the top of the cone are completely negligible.

The angles θ_1 and θ_2 , indicated in Figure 3, are given by:

$$\theta_1 = \tan^{-1} \left(\frac{b}{a+b} \right) \quad (7)$$

$$\theta_2 = \tan^{-1} \left(\frac{c}{h} \right) \quad (8)$$

The solid angle Ω_c subtended by the cone at the location of the small sphere is:

$$\Omega_c = 2\pi (\cos\theta_1 - \cos\theta_2) \quad (9)$$

According to Eq (6) the direct power P_1 from the cone that is captured by the black sphere depends on the solid angle and not on the shape of the cone. Therefore:

$$P_1 = \frac{\Omega_c}{4\pi} \sigma T_c^4 \cdot 4\pi r^2 = 2\pi r^2 (\cos\theta_1 - \cos\theta_2) \sigma T_c^4 \quad (10)$$

where $\Omega_c/4\pi$ is the fraction of the total field of view, seen from the center of the sphere, that is luminous, and $4\pi r^2$ is the surface area of the sphere.

The direct power from the liquid surface, captured by the sphere, is:

$$P_2 = \frac{1}{2} 4\pi r^2 E_L \sigma T_L^4 \quad (11)$$

where E_L is the emissivity of the liquid, and the factor $\frac{1}{2}$ allows for the fact that the liquid occupies $\frac{1}{2}$ of the field of view seen from the center of the sphere.

The power P_3 from the cone reaching the sphere after reflection by the liquid surface equals the power from the cone image, shown in Figure 3, reduced by the reflectance $1-E_L$ of the liquid surface:

$$P_3 = (1-E_L) 2\pi r^2 (\cos\theta_1 - \cos\theta_2) \sigma T_c^4 \quad (12)$$

The total power P_s absorbed by the sphere is the sum of P_1 , P_2 and P_3 . Therefore:

$$P_s = 2\pi r^2 \sigma \left[(2-E_L) (\cos\theta_1 - \cos\theta_2) T_c^4 + E_L T_L^4 \right] \quad (13)$$

The equilibrium temperature T_s of the sphere is obtained by equating the power emitted and the power absorbed:

$$4\pi r^2 \sigma T_s^4 = P_s \quad (14)$$

which gives:

$$T_s^4 = \frac{1}{2} \left[(2-E_L) (\cos\theta_1 - \cos\theta_2) T_c^4 + E_L T_L^4 \right] \quad (15)$$

Table I shows values of P_s and T_s , calculated by Equations (13) and (15), for various values of the height h and the cone temperature T_c , for the following values of the other parameters:

$$T_L = 1700^\circ\text{K}$$

$$E_L = 0.3$$

$$a = 3.8 \text{ cm}$$

$$b = 1.9 \text{ cm}$$

$$c = 3.65 \text{ cm}$$

$$r = 1 \text{ cm}$$

$$\sigma = 5.67 \times 10^{-12} \frac{\text{W}}{\text{cm}^2 \text{ deg}^4}$$

It is seen that P_s and T_s increase quite rapidly with cone temperature T_c , especially for small values of h . This suggests that T_s could be controlled by applying power directly to the cone from an independent RF source. In addition, a light source could be used for fine adjustment of T_s .

The values of P_s and T_s with the cone removed are 89 W and 1058°K, respectively. These values are obtained if one sets $T_c = 0$ in Eq 15.

TABLE I - Variation of P_s and T_s with h and T_c for $T_L = 1700^\circ\text{K}$

h (cm)	θ_1 (deg)	θ_2 (deg)	T_c (oK)	P_s (w)	T_s (oK)
0	26.6	90.0	1500	363	1502
			1600	444	1580
			1700	541	1660
			1800	657	1743
			1900	795	1827
			2000	955	1914
1	21.6	74.7	1500	293	1424
			1600	353	1492
			1700	426	1563
			1800	512	1637
			1900	614	1714
			2000	734	1791
2	18.1	61.3	1500	233	1345
			1600	275	1402
			1700	327	1463
			1800	388	1527
			1900	460	1594
			2000	544	1662
3	15.6	50.6	1500	190	1278
			1600	220	1325
			1700	255	1376
			1800	298	1430
			1900	348	1487
			2000	407	1546
4	13.7	42.4	1500	161	1275
			1600	182	1264
			1700	207	1306
			1800	237	1351
			1900	273	1399
			2000	315	1450
5	12.2	36.1	1500	141	1187
			1600	157	1218
			1700	175	1252
			1800	197	1290
			1900	223	1331
			2000	254	1374

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DIRECT INDUCTION HEATING OF SILICON USING COLD CRUCIBLE TECHNIQUES

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INTRODUCTION

This bibliography lists, in chronological order, references chronicling the developments in the theory, design and usage of RF induction heated cold crucible (skull) melting. No effort has been made to include all the references in the history of the technology, but it is hoped that the majority of the key references are included.

In addition to specifically related references, a limited number of references are included in fields whose technology is relevant to the skull melting process. For this reason, there are included a number of references on levitation melting, plus a few in the fields of direct induction melting and pedestal-pulling using direct induction heating.

The format of the bibliography lists the references in chronological order. Where there is more than one reference for a particular year, the references are divided into three sections. The first section includes all non-patent references; e.g. journal articles, books, U.S. and foreign government reports; the second section includes all U.S. patents, and the third section, all foreign patents. The U.S. and foreign patents are separated because of differences in citation usage. While U.S. patents are cited by publication date, some foreign countries, such as France and Germany cite patents by file date. In this bibliography, both U.S. and foreign patents are cited by publication date.

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II. Design and Construction Techniques

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GROWTH OF HIGH PURITY OXYGEN-FREE SILICON BY COLD CRUCIBLE TECH--ETC(U)

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