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# Low-cost bridgman-type single-crystal growing apparatus

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## **Abstract**

The design, operation, and results obtained using a low-cost, Bridgman-type, single-crystal growing apparatus are presented. The discussion is divided into sections covering the crystallizing crucible, furnace, and sample lowering assemblies along with recommended changes that it was felt would improve the operation.

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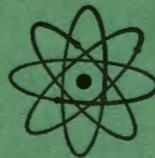


**LOW-COST BRIDGMAN-TYPE SINGLE-  
CRYSTAL GROWING APPARATUS**

By

Edwin H. Olson

**AMES LABORATORY  
RESEARCH AND DEVELOPMENT REPORT  
U.S.A.E.C.**



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LOW-COST BRIDGMAN-TYPE SINGLE-  
CRYSTAL GROWING APPARATUS

By

Edwin H. Olson

August 1960

Ames Laboratory  
at  
Iowa State University of Science and Technology  
F. H. Spedding, Director  
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LOW-COST BRIDGMAN-TYPE SINGLE -  
CRYSTAL GROWING APPARATUS

Edwin H. Olson

ABSTRACT

The design, operation, and results obtained using a low-cost, Bridgman-type, single-crystal growing apparatus are presented. The discussion is divided into sections covering the crystallizing crucible, furnace, and sample lowering assemblies along with recommended changes that it was felt would improve the operation.

Single crystals of zinc, lead, and tin metals, and silver chloride were successfully grown. Sizes of the single crystals varied from  $3/8$  to  $3/4$  of an inch in diameter and were grown in lengths up to  $3\ 1/2$  inches. The crystallizing crucibles used were quartz and graphite, and all had 50 degree conical nucleating tips. Tapered walls on graphite crucibles greatly facilitated the removal of the single crystals from the crucibles.

The equipment was found to be simple to operate and the conditions were readily reproducible. The low cost of approximately \$500.00 should make it of interest to persons desiring to grow their own single crystals of low melting point materials. Dimensioned drawings are included so the equipment can be duplicated.

INTRODUCTION

There is an urgent need for reliable, simple, low-cost, single crystal growing equipment. Physicists, chemists, metallurgists, and others desire single crystals to effectively and accurately

determine various thermal, electrical, chemical and nuclear properties of various materials along the different crystallographic axes. Polycrystalline samples prevent these measurements due to the random order of their crystals.

In the past, investigators desiring to study physical properties of single crystals have had to devise temporary equipment to grow their own single crystals. Many times these investigators spent much time, effort, and money growing their single crystals, but did not describe the equipment or report the operating conditions adequately to permit others to duplicate the equipment and conditions to obtain single crystals. This is understandable since the investigator was more interested in studying and reporting the physical properties of the single crystal than in the growth of the single crystal. For this reason, then, it was felt that an effort should be made to design, test, and report on single-crystal growing equipment so that others could duplicate the equipment and conditions and grow their own single crystals with a minimum expenditure of time, effort and expense.

The primary concern was to determine the crystallization method to be investigated first. Lawson and Nielsen<sup>(1)</sup> and Buckley<sup>(2)</sup> give short descriptions of various single-crystal growing methods. The following methods were considered.

(1) Crystal pulling from the melt.

(2) Bridgman method, consisting of passage of the melt through a steep temperature gradient.

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<sup>1</sup> Lawson, W. D. and Nielsen, S. "Preparation of Single Crystals", (Butterworth Scientific Publications, London, (1958)), Chapter 2, pp 10-30.

<sup>2</sup> Buckley, H. E. "Crystal Growth", (John Wiley and Sons, New York, (1951)), Chapter 2. pp 43-104.

- (3) Growth from the vapor.
- (4) Growth from solution.
- (5) Decomposition and deposition methods.
- (6) Zone melting and crystallization.

The choice of which of the above methods to investigate first was not easy since each could be used in the preparation of many different types of crystals.

Growth of crystals from their vapors would require high temperatures, close temperature control, and the use of a vacuum system, and thus was ruled out as being more complex than desired and probably limited with respect to the number of materials that could be grown into single crystals.

Growth from solutions results in varying amounts of trapped solvents. Therefore, purity would be questionable, and this method was eliminated from consideration.

Decomposition and deposition methods were thought to be limited to too few materials to warrant investigation at this time.

The Bridgman method was finally selected for the first investigations, although crystal pulling, and zone melting and crystallization were also considered. The reasons for this choice were the relatively simple equipment, low cost, and minimum amount of operator attention. Also, the Bridgman unit could be converted to a vertical zone melter and crystallizer simply by providing another short furnace section between the other two furnaces.

The crystal-pulling method does have one advantage in that no crucible has to be removed from the single crystal after it is grown.

In this method the single crystal is formed as it is withdrawn from the melt. This feature should be advantageous for melts that wet the crucible and for materials such as bismuth and antimony which expand upon solidification. The disadvantage of this method is that the single crystal does not have a regular, smooth surface and more cutting and forming operations are required to obtain the shape desired for physical or nuclear studies.

The purpose of this investigation is to design, build, and test a Bridgman single-crystal growing unit and report the operating characteristics, construction details and possible future improvements so that others may duplicate the unit and grow their own single crystals.

It is hoped that this investigation may be one of the first steps toward making the growing of single crystals more of a "science" and less of an "art".

## EQUIPMENT DESIGN

### General Considerations

The first problem was to decide upon the general design of the Bridgman crystal-growing unit and the operating temperature range.

It was decided that the maximum temperature of the furnaces should be around 850°C since, for higher temperatures, conventional thermal insulating materials would probably have to be replaced by more efficient reflecting radiation shields to control the temperature gradient.

Another problem was to determine whether to move the furnaces with respect to the sample or lower the sample through the furnaces. It

was decided that the latter would be simpler. The sample would be suspended from a wire or chain in order to minimize the chance of mechanical shock which might cause new nuclei to form while the single crystal was growing, and result in a polycrystalline sample. With this means of suspension, the sample would hang freely in the center of the furnaces but would not touch the walls as it was lowered from the upper, hot furnace to the lower, colder one.

The next problem was to design a lowering mechanism that would operate at constant speed and yet could be varied over wide ranges. Here it was decided to rely on the constant speed of a. c. induction-type motors and to vary the speed using a variable speed changer and a fixed reduction gear reducer. A pulley, with an attached chain on the output shaft of the fixed gear reducer, would be used to support the sample and lower it through the furnaces according to the speed set on the speed changer. This should fulfill the minimum requirements for a Bridgman-type single-crystal growing apparatus. The photograph in Fig. 1 shows the assembled crystallizer, and Fig. 2 shows the dimensioned drawings. Figure 3 shows the sample lowering mechanism and Fig. 4 the furnace assembly details.

#### Crystallizing Crucible Assembly

The main function of the supporting crucible is to hold the crystallizing crucible as it is lowered through the temperature gradient established by the two furnaces. The supporting crucible must have a low thermal conductivity in order that the horizontal radial heat losses from the

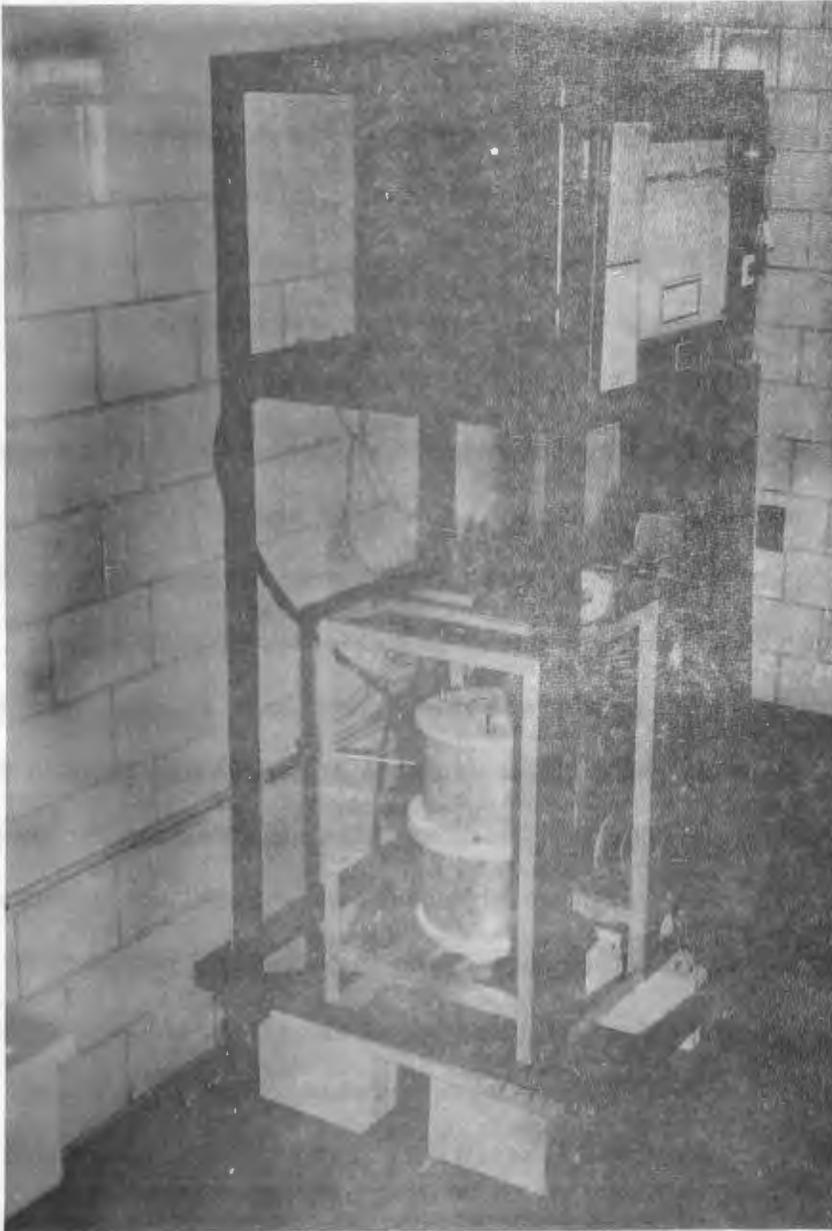


Fig. 1—Photograph of Crystallizer.

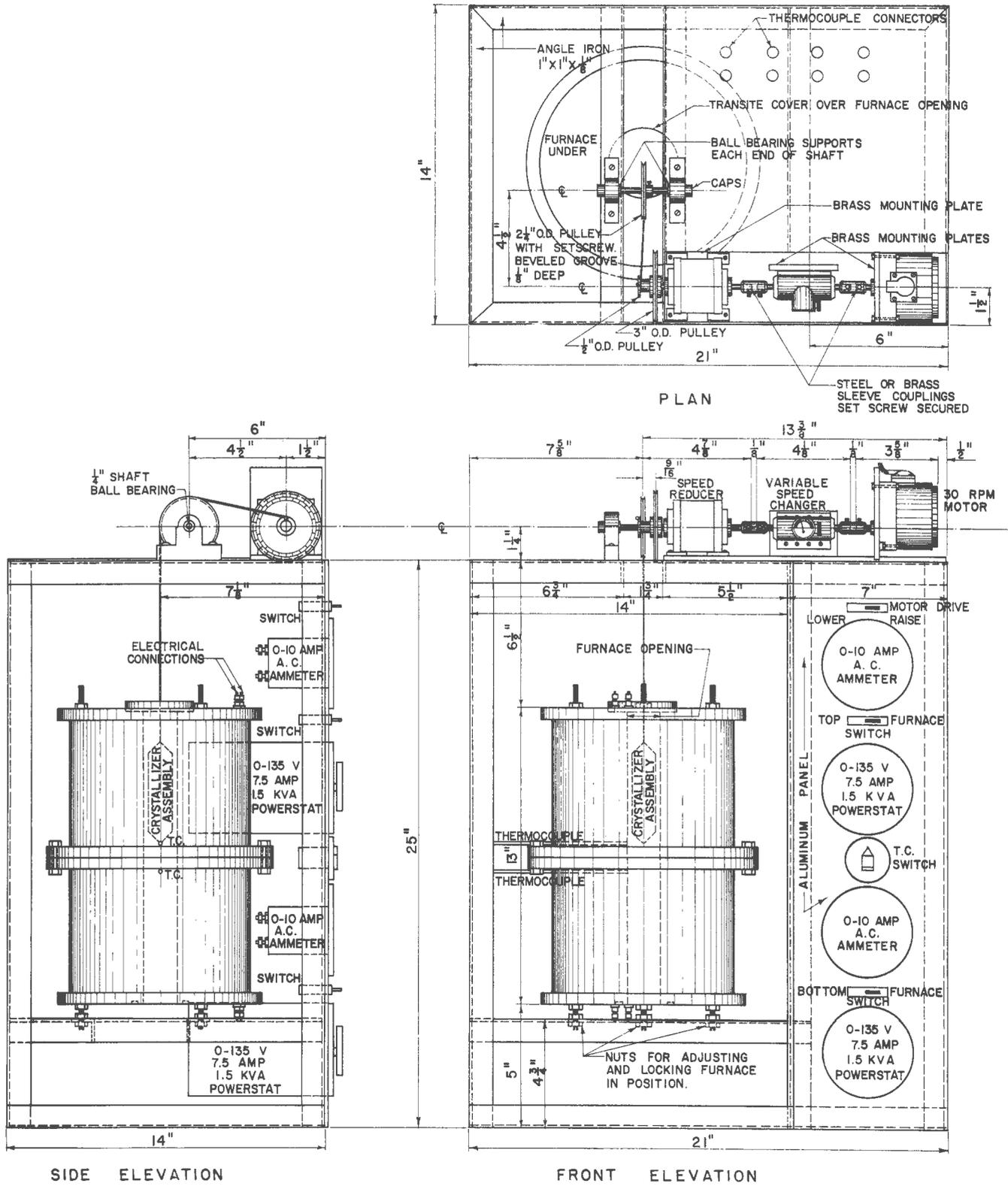


Fig. 2—Dimensioned Drawings of Crystallizer.

## LEGEND

- ① 30 RPM MINNEAPOLIS-HONEYWELL INDUCTION MOTOR
- ② MOTOR-SPEED CHANGER COUPLING
- ③ METRON TYPE 3A SPEED CHANGER  
(5:1 INCREASE TO 5:1 DECREASE)
- ④ SPEED CHANGER-SPEED REDUCER COUPLING
- ⑤ METRON TYPE 10B SPEED REDUCER  
(1936:1)
- ⑥ HIGH SPEED BRASS PULLEY
- ⑦ CALIBRATED SCALE  
(MEASURE VERTICAL TRAVEL OF SAMPLE)
- ⑧ LOW SPEED BRASS PULLEY
- ⑨ SAMPLE LOWERING CHAIN
- ⑩ BRASS IDLER PULLEY
- ⑪ BRASS IDLER PULLEY SUPPORT  
AND BEARINGS

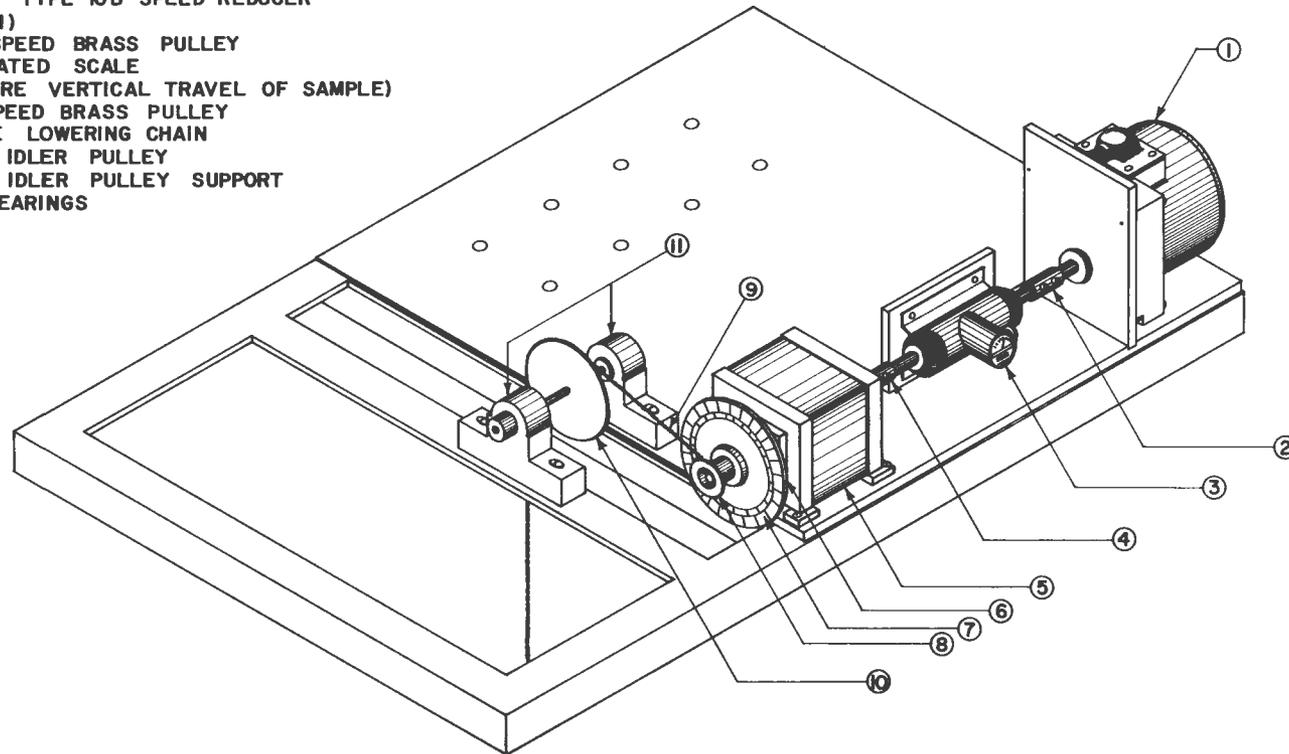


Fig. 3—Isometric View of Sample Lowering Mechanism.

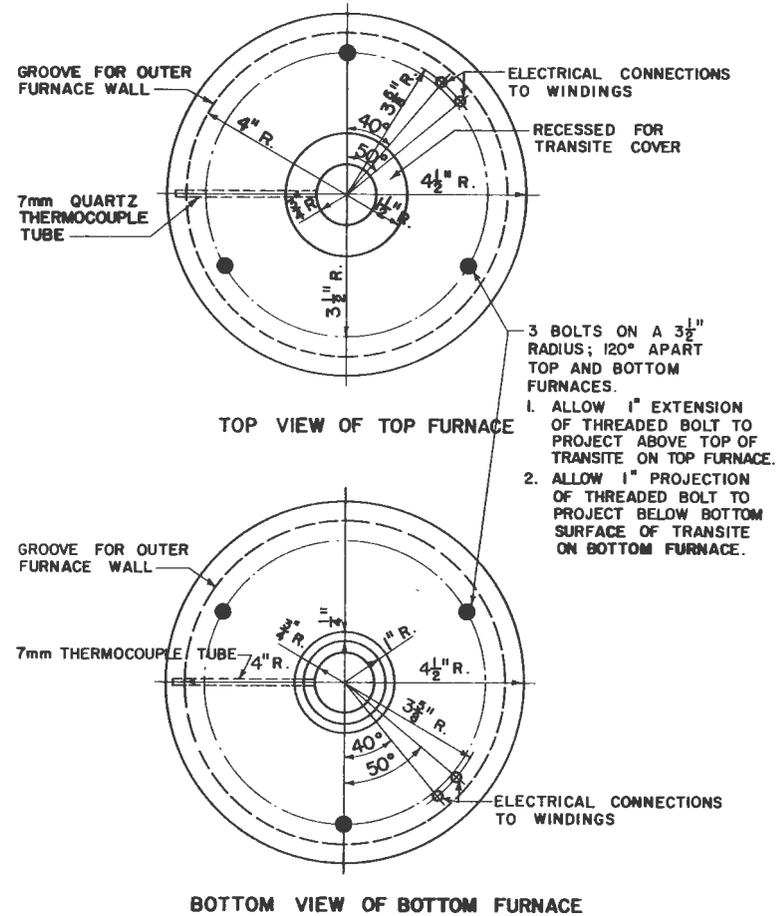
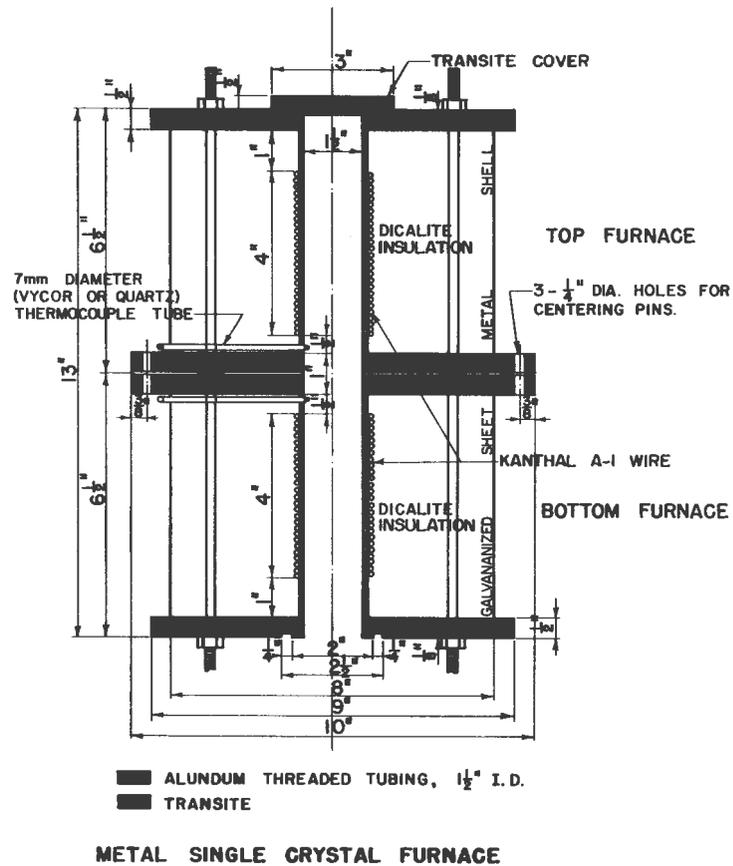


Fig. 4—Dimensioned Drawings of Furnace Assembly.

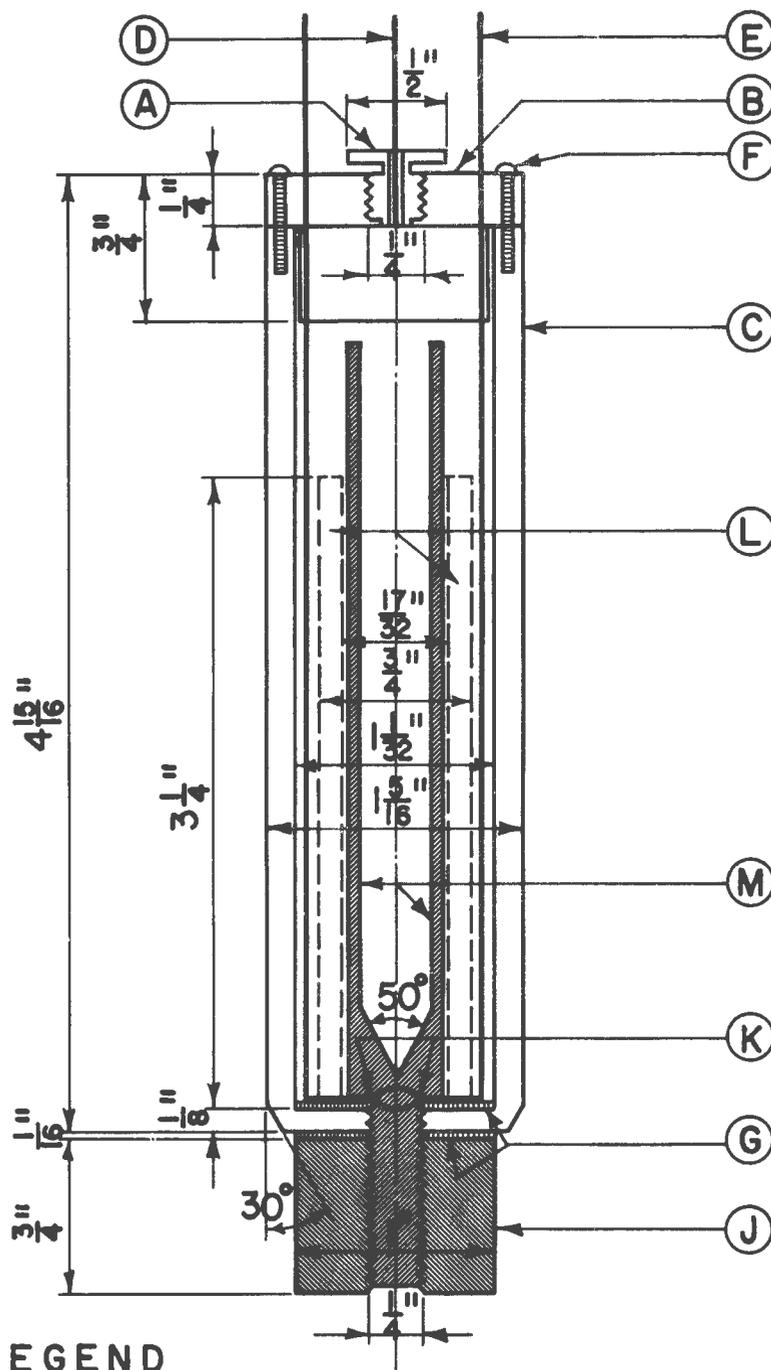
crystallizing crucible are minimized. Excessive heat losses in the horizontal, radial direction would result in a concave crystal-melt interface which would be conducive to the formation of new nuclei at the crucible walls, resulting in the formation of a multicrystal sample.

Two supporting crucible materials were used in these investigations. The first one was quartz and the second one, stainless steel. The stainless steel used was #304, and the quartz was clear fused material. Figures 5 and 6 show the crystallizing crucible assembly and the silver and graphite heat sinks used with the quartz and stainless steel supporting crucibles, respectively. Also shown are the supporting chromel wire, chromel-alumel thermocouple, and the crystallizing crucibles.

One disadvantage of stainless steel, as compared to quartz, is its higher thermal conductivity which would result in greater horizontal, radial heat losses from the crystallizing crucible. To counteract this effect, asbestos and alundum insulation with very low thermal conductivities were used.

The graphite and quartz crystallizing crucibles tested, shown in Fig. 7, had 50 degree (included angle) conical tips. The function of the tips was to reduce the number of nuclei formed and to permit rapid growth to occur during the first part of the crystallizing run. This rapid growth should permit the fastest growing nucleus to crowd out all of the others in the cone tip area and result in one single crystal continuing to grow as the sample was lowered. Theoretically, the graphite crucibles should

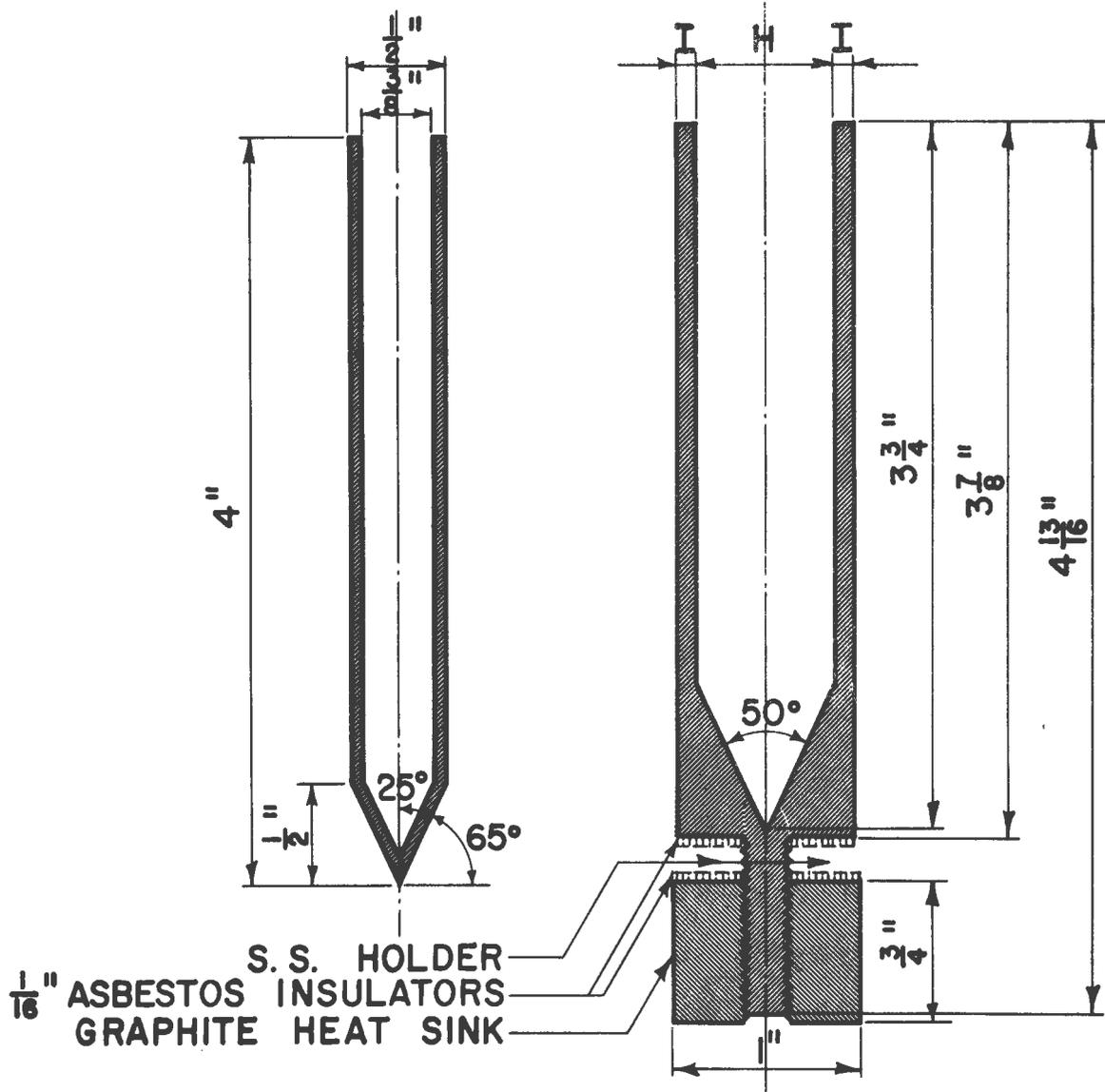




### LEGEND

- (A) SUPPORTING WIRE LOCK (SS No. 304)
- (B) SUPPORTING CRUCIBLE TOP (SS No. 304)
- (C) SUPPORTING CRUCIBLE CASE (SS No. 304)
- (D) CHROMEL SUPPORT WIRE (22 GA.)
- (E) CHROMEL-ALUMEL T.C. (22 GA.)
- (F) No. 2 - 56 S.S. SCREWS
- (G) ASBESTOS INSULATOR (1/16" THICK)
- (J) GRAPHITE HEAT SINK
- (K) THERMOCOUPLE JUNCTIONS
- (L) ALUNDUM TUBE
- (M) TYPE B CRYSTALLIZING CRUCIBLE

Fig. 6—Stainless Steel Crystallizing Crucible Assembly.



TYPE A                      TYPE B  
 (GRAPHITE AND QUARTZ)      (GRAPHITE ONLY)

DIMENSION	CRUCIBLE SIZE		
	SMALL	MEDIUM	LARGE
H	3/8"	9/16"	3/4"
I	1/16"	3/32"	1/16"

Fig. 7—Graphite and Quartz Crystallizing Crucibles.

be better than quartz due to the much higher thermal conductivity of graphite. Since the thermal conductivity of graphite is higher than that of many materials, the heat of crystallization would be removed quite rapidly by transfer to the graphite heat sink below the crucible.

The graphite and silver heat sinks were used to provide a low-resistance path for vertical transfer of heat and thus help establish a steep vertical temperature gradient. This should help maintain the desired planar crystal-melt interface and suppress the formation of new nuclei .

A 24 gauge chromel supporting wire was selected because of its high temperature oxidation resistance and low cross-sectional area available for heat losses by conduction up the wire. The 24 gauge, chromel-alumel thermocouple wires were selected for the same reasons.

The thermocouple actually consisted of two junctions made by wrapping each of the wires 180 degrees around a cylindrical mandrel and then wrapping the end of each wire tightly about the other to give a good electrical contact. This resulted in two thermocouple junctions connected in parallel so that an average temperature reading of the two junctions would be obtained. Note in Fig. 6 that these two junctions, shown at K, are in the same horizontal plane and at the same radial distance from the melt which is to be grown into a single crystal. Therefore, the same temperature should exist at both points and there should be no error resulting from the averaging of the electric potentials developed at both junctions to give the average temperature. The reason for this particular construction was to ensure that the thermocouple

would be at the same position on duplicate runs and that it could be used to check the reproducibility of temperatures for a given sample-lowering speed and furnace current setting. Also, if one of the thermocouple wires should break between the two junctions during a run, the other junction would continue to operate and indicate the temperature.

### Furnace Assembly

The furnaces were fabricated and assembled by our own shops rather than purchased from commercial suppliers. This was done for reasons of compactness and provisions for furnace thermocouples. The furnace assembly and construction details are shown in Fig 4. As can be seen from the drawings, the construction is quite simple and can be made with a minimum of shop tools and equipment.

The two furnaces are mirror images of each other and consist of the following:

- (1) A threaded alundum core wound with 20-gauge Kanthal A-1 wire.
- (2) Transite end pieces to hold the alundum core in place and to hold the sheet metal sides containing the Dicalite high-temperature insulation.
- (3) Horizontal quartz tubes to provide for the thermocouples used to measure the temperatures at the bottom of the upper furnace and the top of the lower furnace.

Auxiliary equipment used in conjunction with the furnace assembly was:

- (1) Powerstats to control the current flow to each furnace.
- (2) Ammeters to indicate the amount of current flowing through each furnace.

- (3) Double-pole switches to turn on or shut off the power to the furnaces while inserting the supporting crucible assembly prior to a run or when working on the crystallizer.

Alundum was chosen for the furnace core material because of its high-temperature stability and its thermal shock resistance.

Kanthal A-1 wire was chosen because of its characteristic high-temperature resistance.

Dicalite was used because of its high-temperature insulating properties.

Two furnaces were used, instead of one furnace with two separate sets of windings, for the following reasons:

- (1) A steeper vertical temperature gradient could be obtained using two furnaces since there would be no common alundum core to transfer heat by conduction. This would also permit the insertion of various gasket materials if they were needed.
- (2) The distance between the windings could be varied simply by inserting varying thicknesses of insulating materials.
- (3) The Bridgman crystallizing furnace could easily be converted to a vertical-zone refining unit by the insertion of a short, vertical, high-temperature furnace between the two present furnaces.

The top split-transite furnace cover was designed in two, overlapping, slotted pieces to prevent excessive loss of heat from the furnaces, yet permit easy, vertical movement of the supporting crucible wire and thermocouple wires as the sample was lowered through the furnaces.

The transite cover for the lower furnace shown in Fig. 2, is separated from the transite end-plate of the furnace by the 1/4 inch thickness of the taps on the furnace bolts. The transite cover is the same diameter as the furnace end-plate to prevent drafts from removing varying amounts of heat from the furnace assembly. A slight amount of heat removal by natural vertical convection of air through the furnace core, would be desirable to help maintain a steep, vertical temperature gradient. This cooler air would be continually flowing around the graphite and silver heat sinks, promoting steeper, vertical temperature gradients in the crystallizing crucible.

No voltage stabilization of the electric current supply to the furnaces was provided since the voltage supply available was reported to be quite constant. Automatic control of the temperature was not provided since it was desired to keep the equipment-cost low.

The main functions of the furnace thermocouples were to give an indication of the vertical temperature gradient and to determine when the furnaces were at steady-state temperatures prior to the start of a crystallization run. In replicate runs, they served as checks on the reproducibility of the temperature conditions with the furnace currents set at the same values as were used in previous crystallizing runs.

In the initial runs using the quartz supporting crucible, an asbestos gasket was placed between the two furnaces. In the center of the asbestos was a 1 inch diameter hole. This permitted the 3/4 inch diameter quartz supporting crucible to pass through but kept to a minimum the free area

for convective mixing of the hot, upper-furnace air with the cooler, lower-furnace air. In the runs using the 1 5/16 inch diameter stainless steel supporting crucible, no gasket material was needed since the annular space between the crucible and the furnace wall was only 3/32 of an inch.

### Sample-Lowering Assembly

The sample-lowering assembly, as shown in Fig. 3, consisted of the following units:

- (1) A constant speed, 30 RPM, 110 volt, 60 cycle, Minneapolis-Honeywell, recorder chart drive induction motor.
- (2) A capacitor-powerstat electric hook-up with the motor, to permit a reduction of the motor speed. (Not shown)
- (3) A variable speed changer to give any desired sample-lowering speed within the limits of the speed changer.
- (4) A fixed speed reducer to reduce the speed range to values usable in growing single crystals of various materials.
- (5) A pulley secured to the output shaft of the speed reducer. A chain attached to the pulley to translate the rotational motion to a vertical motion necessary to lower the crystallizing assembly through the furnaces. An idler pulley is positioned such that the chain hangs directly over the center of the furnace core.
- (6) A reversing switch to change the direction of rotation of the drive motor.

Perhaps the most important unit in this assembly is the constant speed induction motor, since constant sample-lowering speed is one of

the more essential factors in growing single crystals by the Bridgman method. Most power and utility companies supply a constant frequency alternating current; therefore an induction motor, which runs at a constant speed provided the frequency of the current is constant, is used. This constant, rotational speed can then be easily converted to a constant, vertical, lowering speed by the use of a speed changer, speed reducer, and the pulley and chain arrangement mentioned above.

The capacitor-powerstat arrangement, shown schematically in Fig. 8, for reducing the speed of the induction motor, has been previously used by others in this laboratory. This control of the speed, however, is attained at the sacrifice of output torque of the motor; also, a speed reduction of one half is all that can be accomplished with the maintenance of a constant speed.

The reversing switch, to change the direction of rotation of the motor, was found to be quite useful in vertically positioning the sample, and for short preliminary trials to determine the lowering speed prior to the crystallizing run.

The variable speed changer was placed between the drive motor and the fixed speed reducer because of the low torque requirements ahead of the 1936:1 speed reducer. Metron variable speed changer type 3A and Metron-type 10B fixed speed reducer were selected because of their compactness and high torque ratings. The operating range of the speed changer was from a 5:1 increase to a 5:1 decrease or a 25-fold change in speed.

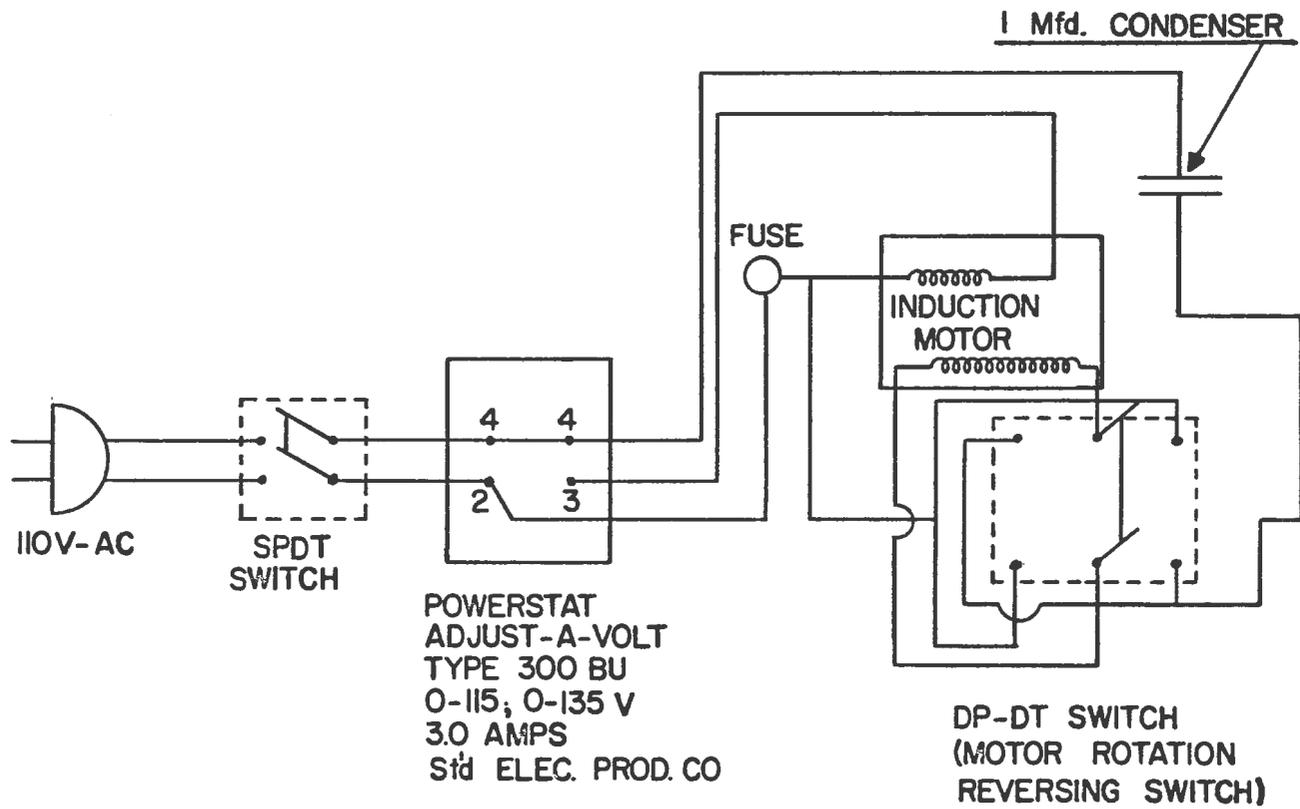


Fig. 8—Schematic Diagram of Method for Reducing the Speed of the Sample

Lowering Motor.

A chain, rather than a wire, was used on the pulleys so that the sample, as it was being lowered, was always suspended in the center of the furnace. A chain conforms to the contour of the pulleys while a wire does not, unless a large weight is attached to keep it stretched tight. The use of a large weight was not desirable since the supporting wire might then be susceptible to stretching and possible failure as it passed through the hot zone of the upper furnace.

The use of different diameter pulleys permitted the lowering speed and ranges of lowering speed to be varied. In these investigations 1/2 inch diameter and 3 inch diameter pulleys were used which permitted a six-fold change in vertical lowering speed. The range of vertical lowering speeds attainable with this assembly, ranges from approximately 0.005 inches per minute to 0.75 inches per minute without using the capacitor-powerstat method to reduce the speed of the induction drive motor. The use of this method to reduce the speed of the motor permits lowering speeds as low as 0.0025 inches per minute.

#### Other Components

The recording of temperatures was by a Bristol, adjustable zero-adjustable range, potentiometric, single pen recorder. This unit was checked periodically using a Leeds and Northrup hand potentiometer. The Bristol recorder was operated with a 10 millivolt scale span over a distance of 11 inches, which permitted the temperature to be read to the nearest 1/2°C without difficulty.

All three thermocouples, the sample, the top furnace, and the bottom furnace, were connected to the recorder through a rotary switch so that

the temperature at any of these three positions could be recorded by switching to the appropriate position.

Calibration scales were made and attached to the pulleys, secured to the output shaft of the fixed speed reducer, so that the vertical distance traveled by a sample during a crystallizing run could be determined quickly by reading the attached calibrated scale. These scales were also useful in determining lowering speeds at various time intervals during the run, to determine the constancy of the vertical lowering speed.

Two pieces of plexiglass with guide slots were used to guide the thermocouple wires vertically and prevent dragging or binding as they passed through the small holes in the split transite cover for the top furnace.

#### MATERIALS CRYSTALLIZED

The materials used in testing the operation of this crystallizer were the low melting, generally non-reactive metals: zinc, lead, and tin, and thallium and one inorganic salt, silver chloride, all of which have melting points below 500°C. The thallium metal and silver chlorides were used in the tests because other members of this laboratory were interested in studying their physical properties. The thallium was unique among these materials inasmuch as it was the only one showing a phase change in the solid state. The melting point of thallium is reported in the handbooks as 305°C; and upon freezing, thallium has a cubic structure. As the temperature is reduced below 225°C, the cubic transforms into the hexagonal form. This transformation reportedly occurs quite rapidly.

The materials were not of ultra-high purity since it was felt that the less pure ones would be satisfactory with respect to evaluating the performance of the crystallizer. Purities of the materials were generally greater than 99.98% with respect to foreign metals.

The zinc used was "Bunker Hill" slab zinc, electrolytic grade, supplied by Sullivan Mining Co. of Silver King, Idaho. Purity certified and stamped in the slab was 99.99+%.

The lead used was Fishers "Reagent" grade granular material, 99.98+% purity.

The tin used was the mossy form of Baker's "Reagent" grade, 99.999+% purity.

The thallium was supplied by Robert Meyerhoff of this Laboratory, who reported that it was obtained from American Smelting and Refining Co. in the form of cast stick. Its purity was reported to be approximately 99.95+%.

The silver chloride was supplied by Dr. Richard Reade of this Laboratory. Dr. Reade prepared this material from silver nitrate and ammonium chloride and purified it using a cyanide treatment. The purity was not established but probably was greater than 99.9%.

#### EXPERIMENTAL PROCEDURE

The general procedure used in the test runs consisted of the following three steps:

- (1) Preparation of the sample of the material to be crystallized.
- (2) Making the crystallization run.
- (3) Sample removal and testing to determine whether or not a single crystal was obtained.

### Sample Preparation

The method of preparation of the sample depended upon the form in which the sample material was available. The zinc was available in slab form, and cylindrical slugs with 50 degree conical tips were machined from the slab. The granular lead and mossy tin were both melted in air and cast into the desired diameter slugs with 50 degree conical tips, using one of the graphite crystallizing crucibles. The thallium was already in rod form but was swaged to reduce it to the proper diameter. In the case of thallium, no 50 degree conical tip was used on the initial sample. The silver chloride was precast directly into the graphite crystallizing crucible, starting with purified, powdered material.

All of the metal samples were etched with appropriate etching reagents to remove any surface oxide film before placing them in the crystallizing crucible.

The etching reagents, used to remove surface oxide films and also to bring out grain boundaries in the crystal structure, were as shown in Table I. The etched polycrystalline samples were thoroughly washed with distilled water to remove traces of salt formed by reaction of the metal with the acid, and the sample was dried at room temperature before it was placed in the crystallizing crucible.

### Crystallization Run Procedure

The steps involved in making a crystallization run are as follows:

- (1) Assembly of the crystallizing unit.
- (2) Insertion of the crystallizing assembly into the furnace and attaching it to the chain of the lowering mechanism.

Table I  
Etching Reagents Used for Metals

Metal	Etching Solution	Time
Zinc	3 parts conc. HCl 1 part dist. water	10-20 sec.
Lead	20 ml acetic Acid 27 ml nitric Acid 106 ml dist. water	30 sec-2 min. (Longer for developing good grain boundaries)
Tin	(a) conc. HCl (b) 1 volume conc. HCl 1 volume dist. water	10 sec. 20 sec-1 min.
Thallium	conc. H <sub>2</sub> SO <sub>4</sub> for followed by dist. water for	10 sec. approx. one hour

- (3) Positioning of the crystallizing assembly in the top furnace.
- (4) Heating up of the furnaces and melting of the sample.
- (5) Waiting period for the sample, top and bottom furnace temperatures to reach steady state readings.
- (6) Lowering of the sample through the furnaces to grow the single crystal.
- (7) Furnace cooling period before removing the crystallizing unit from the furnace.

The assembly of the crystallizing unit involved the placing of the etched, washed, and dried sample into the crystallizing crucible. The crystallizing crucible was then placed in the supporting crucible case to which the top containing the chromel supporting wire was then attached. The same procedure was used with both the stainless steel and quartz supporting crucible-crystallizer assemblies. Reference to Figs. 5 and 6 should answer any questions as to the method of assembly of the crystallizing units.

The crystallizing assembly was then inserted into the top furnace, and the chromel supporting wire was attached to the chain connected to the lowering mechanism. The split transite cover for the top furnace was then placed in position around the thermocouple and chromel supporting wires, and the crystallizing assembly was raised until it rested immediately below the top transite furnace cover. The pulley securing the chain to the output shaft of the speed reducer was then secured by tightening the allen set screw. The sample thermocouple wires were then attached to the rotary switch leading to the potentiometer recorder.

The current to both furnaces was turned on. Three amperes of current were used initially to heat both furnaces rapidly to the desired operating temperatures for the crystallizing run. The current to the bottom furnace was reduced first since it was always carried at a lower temperature than the top furnace. The top furnace current was not reduced until the sample thermocouple indicated that the temperature was approximately 80 degrees above the melting point of the sample. The current was then reduced, and the temperature of the sample was allowed

to fall to between 30 to 50 degrees above the freezing point. When all temperatures were stabilized, that is, showed less than a 2 to 3°C change in 15 minutes, the drive motor of the lowering mechanism was turned on and the sample lowered through the furnace to grow the single crystal. At this point, a fairly close record was kept of the sample thermocouple temperatures since it was this record that was used to determine the vertical temperature gradient that the sample passed through during the run.

#### Testing of Samples

After the crystallizing run, the furnaces were turned off, and the sample was generally allowed to cool to room temperature overnight. In some runs the sample was removed from the furnace, after a short cooling period, in order that another run could be made the same day. The cooled crystallizing crucible was then removed and inverted, and the crystal sample was removed by gentle tapping.

In the case of thallium, portions of the sample adhered tightly to the graphite crucible. The crucible had to be mechanically removed using a coping saw and sharp knife and being careful not to bend or deform the soft thallium sample. For lead grown in a quartz crucible, it was found that the lead stuck very tightly to the quartz and could not be mechanically removed without deforming the sample. Therefore, the rest of the runs were made using graphite crystallizing crucibles to which the lead did not adhere.

In the case of silver chloride, the graphite was removed from the sample by machining in a lathe since graphite was strongly wetted by the molten silver chloride.

After the samples had been separated from the crystallizing crucible, they were etched using the solutions given in Table I, to determine the presence or absence of grain boundaries. The presence of grain boundaries, of course, was definite proof that a single crystal was not obtained. For zinc, after etching had failed to develop any grain boundaries, the sample was twisted near one end using 2 pairs of pliers to develop a cleavage plane perpendicular to the "c" axis. The mirror-like surface of the cleavage plane was additional evidence that the sample was a single crystal.

In several instances the samples were also checked by taking a series of 3, Laue, back reflection, x-ray pictures to ensure that the samples were truly single crystals. The x-ray pictures were generally taken near the cone tip end, the opposite end and near the middle after rotating the sample 180 degrees about its longitudinal axis. By proper positioning, the pattern of spots on all three pictures should be superimposable if the sample were a single crystal.

The etching and polishing of the silver chloride samples were performed by Dr. Richard Reade<sup>(3)</sup> of this Laboratory. Following is the procedure that he used.

The polishing was accomplished by hand, using a piece of Buehler "microcloth" mounted on a flat glass plate. The crystal was mounted in a holder, using Duco cement, and then ground on a piece of 600 grit silicon carbide paper. The sample was then polished on cloth using a paste mixture of "Vel" detergent, water, and enough 600 grit silicon

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<sup>3</sup> Dr. Richard Reade--Private communication.

carbide to make a smooth paste. For the final polish the crystal was adjusted in the holder so that the crystal surface barely touched the polishing cloth. The silver chloride was etched by treating it with concentrated ammonia to render the surface opaque. Water was then added, drop by drop, until the sample became transparent. The sample was then removed from the ammonia solution, rinsed well with distilled water, and permitted to dry. The dry, polished, and etched surface was then inspected by light reflection from the surface to determine whether any crystal boundaries were present. The absence of grain boundaries was accepted as proof that the sample was a single crystal.

#### EXPERIMENTAL RESULTS

Since the purpose of this paper is to describe the operation and functioning of the equipment, the discussion with respect to the actual crystal growth conditions for the various materials tested will be held to a minimum.

First of all, this Bridgman crystallizing unit functioned quite satisfactorily in growing single crystals of zinc, lead, and tin. There are certain changes in the design that, it is believed, would improve the performance. These changes will be discussed under the following headings:

- (1) Crystallizing crucible assembly.
- (2) Furnace assembly.
- (3) Sample-lowering assembly.

### Crystallizing Crucible Assembly

The design of this unit was apparently quite satisfactory with respect to the use of graphite crucibles since single crystals could be grown over a wide range of lowering speeds. Using 3/8 inch diameter crucibles, zinc single crystals could be grown at speeds ranging from 0.024 to 0.75 inches per minute with a vertical temperature gradient of approximately 12°C per centimeter. At the higher speeds the crystallization took place much lower in the furnaces. Also, at the higher lowering speeds the apparent vertical temperature gradient, as determined from the sample thermocouple readings, was much lower, indicating that the horizontal temperature gradient must have been much higher. The horizontal insulation, consisting of an alundum tube, an air space, and an asbestos sheet, was apparently effective in keeping the horizontal heat transfer low enough to prevent the formation of a concave solid crystal-melt interface which would have permitted new crystal nuclei to grow at the walls and a polycrystal or multicrystal to grow.

The use of quartz crystallizing crucibles was not as satisfactory as graphite. This was due to the fact that the thermal conductivity of graphite is nearly a factor of 100 higher than the quartz. The graphite, therefore, readily transmitted the heat of crystallization vertically through the heat sink to the colder furnace below the sample, while the quartz presented high resistance to the vertical heat flow. Therefore, slower lowering speeds were required when using quartz crystallizing crucibles to permit the heat of crystallization to be dissipated vertically instead of horizontally.

Gaskill<sup>(4)</sup> reported that single crystals of zinc could be grown in quartz crucibles at a rate of 0.02 inches per minute with a temperature gradient of 12°C per centimeter and that polycrystalline samples resulted when the speed was increased to 0.05 inches per minute. In this investigation it was found that single crystals of zinc could be grown in quartz at 0.049 inches per minute with a vertical temperature gradient of 17.3°C per centimeter. The diameter of Gaskill's samples was approximately 1/4 inch, while that of the samples used in this investigation was approximately 3/8 inch. Therefore, at the same vertical lowering speed, the 3/8 inch samples represented twice as much zinc crystallizing per unit time as was crystallized using the 1/4 inch samples with twice as much heat of crystallization to be dissipated. We were able to produce single crystals of zinc at higher rates presumably due to the insulation provided to reduce the horizontal heat losses during a crystallizing run.

Another disadvantage of quartz crucibles was that they often had to be sacrificed after each run since slight irregularities in the tubing diameter prevented the single crystals of zinc from being easily removed without breaking the tube. In the case of lead, only one run was made in quartz. It was found that the lead thoroughly wet the quartz making it impossible to remove the quartz without deforming the sample. No difficulty was experienced when the graphite crucibles were used to grow single crystals of zinc, lead, and tin. These single crystals were easily

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<sup>4</sup> Gaskill, J. R. "A Modified Bridgman Furnace for the Growing of Zinc Monocrystals", Livermore Research Laboratory Report No. LRL-126, California Research and Development Co. (April-1954).

removed by inverting and gently tapping the crucible. Late in the investigations, graphite crucibles with  $1/2$  and  $1$  degree tapered walls were used, and this greatly facilitated the removal of the single crystals. Generally, simply inverting the crucible resulted in the removal of the sample. It is believed that a taper of as little as  $1/4$  degree would have been satisfactory.

Thallium metal and silver chloride both adhered strongly to the walls of the graphite crucibles, and the crucibles had to be sacrificed to recover the crystallized samples. For thallium it is believed that an oxide film was responsible for the adhesion to the crucible walls since portions of the sample were free and did not stick to the walls. In the areas that were stuck it was noted that there was also a brown film which was assumed to be thallic oxide. In the case of silver chloride, this molten salt thoroughly wetted the graphite crucible walls, filling the voids in the graphite quite completely. In one run where the silver chloride had been melted and held in the molten state overnight in the crucible, complete penetration of the walls occurred with resultant leakage into the crystallizing furnace during the single-crystal growing run.

The primary advantage of quartz crucibles is that they can be evacuated and then sealed off in cases where atmospheric oxidation is not desired or cannot be tolerated. Another advantage is that, at higher temperatures, the quartz will withstand the oxidation of the atmosphere while the graphite will not.

The  $50$  degree included angle tip used on all crucibles appeared to be quite satisfactory in permitting the fastest growing nuclei formed in

the tip, to grow and crowd out all of the other nuclei formed. Close inspection of the single-crystal samples failed to show, at the tip, any grain boundaries that were visible to the naked eye. The nuclei formed must have been very small and the rapid growth of the fastest growing nucleus must have rapidly overtaken and crowded out the other nuclei. Also, the etching procedure probably removed many of the small grains that were present on the outer surface at the tip.

The single crystals grown were not crystallographically oriented since no provisions were made to grow from an oriented seed. The cleavage planes in the zinc sample runs were measured using a protractor to determine whether there was any particular preferred orientation of the samples with respect to the cylindrical axis. Nineteen samples grown in graphite crucibles were checked and six samples grown in quartz were measured. The average angles of inclination of the "c" axis of hexagonal zinc with the cylindrical axis of the samples grown in graphite and quartz were 55.1 and 54.7 degrees, respectively. The distribution of values appeared to be random and did not tend to cluster about the 50 to 55 degree angle. Values ranged from 13.5° to 87.5°. Thus, there does not appear to be any preferred crystallographic orientation due to the crucible material or angle at the tip.

The #304 stainless steel supporting crucible performed very satisfactorily and was used for more than 40 crystallization runs in addition to approximately 15 test runs to determine temperature gradients prior to making the crystallization runs. The worst attack came from one run in which the molten silver chloride leaked and severely corroded the surface

of the unit. In one trial run the temperature was raised to 800°C and held for approximately one hour. The center portions of the crucible, which were the hottest and probably closer to 900°C than the 800°C temperature recorded at the bottom of the unit, scaled considerably on the outside due to atmospheric oxidation. If #347 stainless steel had been used, the unit would probably have withstood the atmospheric oxidation more satisfactorily. Nickel or inconel would probably be satisfactory alternate materials of construction for use in fabricating the supporting crucible assembly. It was also noted in this high temperature test run, that the graphite crystallizing crucible inside of the stainless steel supporting crucible withstood the air oxidation. The graphite heat sink screwed on the bottom of the graphite crucible was badly oxidized and would not have withstood a normal length crystallizing run. If this unit is used for high temperature crystallizing runs, this graphite heat sink should be replaced by one made of silver or other non-reactive, high melting material with high thermal conductivity.

The chromel-alumel thermocouple performed quite satisfactorily even though only twisted wire, unwelded junctions were used. The sample thermocouple, located very close to the 50 degree nucleating tip, was useful in indicating the position in the furnace that the sample began to crystallize. The lag in temperature noted for the sample thermocouple was generally between 5 and 10°C as evidenced by the melting and freezing of lead, tin, thallium, zinc, and silver chlorides during crystallizing runs. This lag for each individual material was nearly the same for different diameter graphite crucibles. The

thermocouple construction of the sample thermocouple, whereby the cone point or threaded rod from the base of the graphite crucibles rested directly against the two thermocouple junctions, was a very good method of keeping the thermocouple at the same position on successive runs. Also, the thermocouple was in contact with the crystallizing crucible in all runs and therefore, was capable of giving good, reproducible readings since there was no high-resistance, thermal air gap between the thermocouple and the crucible. The three thermocouples which were provided were sufficient to determine the conditions that should be used for crystallizing single crystals of the materials tested. It would have been helpful to have had another thermocouple attached to the top of the crystallizing crucible in order to determine when the last of the sample had crystallized.

In order to determine the effect of lowering speed and presence of zinc as compared to the absence of zinc, a series of runs were made using the same furnace power settings and the same crystallizing crucible assembly consisting of the stainless steel crucible holder and 3/8 inch diameter graphite crystallizing crucible (type B), both of which were shown in Fig. 5. At the start of the actual crystal growing runs, the furnace power settings were 2.13 and 0.82 amperes for the top and bottom furnaces, respectively. The results of these runs are shown in Fig. 9 where the temperatures, as measured by the sample thermocouple, are plotted vs. the distance that the sample traveled vertically to the colder, lower, furnace. It will be noted by comparing Runs A and 2 that the presence of molten zinc in the

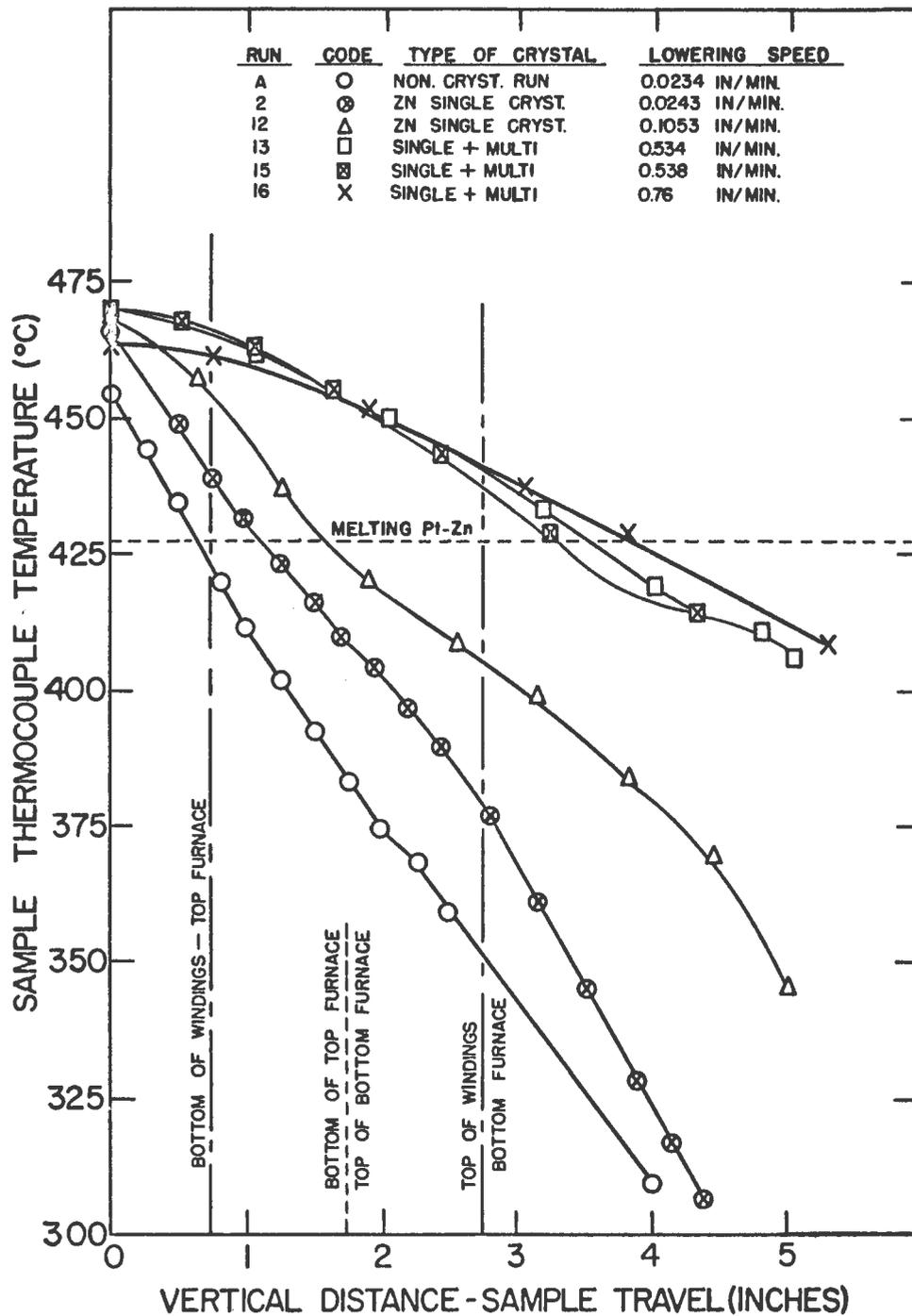


Fig. 9—Graph of Sample Temperature vs. Position in the Furnace Assembly for Zinc Single Crystal Runs.

crystallizing crucible of Run 2 resulted in a higher starting temperature and lower temperature gradient ( $^{\circ}\text{C}/\text{inch}$ ) in the range from  $450^{\circ}\text{C}$  down to  $420^{\circ}\text{C}$ . Both of these effects are probably due to the fact that the molten zinc has a much higher thermal conductivity than the air, thus resulting in a lower resistance to the transfer of heat from the hot, upper part of the graphite crystallizer to the colder area at the tip and graphite heat sink below.

The effect of increasing the lowering speed was to decrease the apparent vertical temperature gradient as recorded by the sample thermocouple. As was expected, the vertical heat transfer is limited to passage through the  $1/4$  inch diameter stem of the crystallizing crucible to which is attached the graphite heat sink. Therefore, increasing the speed results in a higher temperature at the tip because of the smaller time interval to dissipate the heat through the small  $1/4$  inch diameter graphite stem to the heat sink. This resulted in the freezing plane being located at lower positions in the cold furnace, on those runs made using higher speeds. In Runs 13, 15 and 16, shown on Fig. 9, a single crystal was nucleated and grown from the tip, but limitations of the equipment prevented lowering the sample sufficiently so that the whole sample could be passed through the freezing plane. This, then, resulted in the formation of a multi-crystal sample when the lowering was stopped and the furnace power turned off.

The graphite heat sink attached to the graphite crystallizing crucibles was very effective in keeping the sample centered in the supporting crucible and in maintaining good contact of the sample thermocouple with the crystallizing crucible. It also ensured that the

thermocouple was in the same position for replicate runs, permitting temperature comparisons to be made on runs using the same furnace settings. This was quite useful in determining the effect of increasing speed on the vertical temperature gradient as recorded by the sample thermocouple.

The chromel supporting wire performed very satisfactorily. It was necessary to keep it straight and in a perpendicular position with reference to the stainless steel cap of the supporting crucible in order to maintain the crystallizing crucible assembly in the center of the furnaces. A stainless steel or inconel chain would also have been satisfactory provided it had ample strength at the temperatures used to resist stretching.

#### Furnace Assembly

There were no major complaints on the furnace assembly. A minor change to be made when building a new pair of furnaces would be to increase the height of each by 1 to 2 inches. This would permit the growth of longer samples and also would probably result in a more uniform temperature along the heated length of each furnace. It was noted, when the  $3/4$  inch diameter samples were melted, that apparently the top of the solid sample melted last since the temperature measured by the sample thermocouple at the tip showed two decreases in temperature during the 6 to 9 minute period required to completely melt the sample. Apparently the bottom of the sample melted first and then heated up before the solid, top part of the sample settled to the bottom

and melted. A longer furnace would probably have resulted in the sample melting from the top down. The larger furnaces would have had a larger capacity to store heat, making them more insensitive to voltage supply variations and resulting in greater temperature stability. This feature, however, would not have been of importance in the runs made in these investigations except for the very slow speed runs made at 0.0025 inches per minute. An alternative solution to the problem of voltage supply variation would be to provide for either voltage stabilization or automatic control of each furnace temperature.

The Kanthal windings, alundum core, Dicalite insulation, transite end pieces, and furnace end covers performed quite satisfactorily.

In building new furnaces it would be advisable to provide for movable thermocouples inside of the top and bottom furnace cores in order to permit a more accurate evaluation of the actual vertical temperature gradient in the furnace assembly at steady state temperature conditions. As mentioned previously, the sample thermocouple does not give a true vertical temperature gradient since the heat of crystallization passes by this thermocouple and the indicated gradient changes with the speed of crystallization.

#### Sample Lowering Assembly

This unit performed exceptionally well and there are no recommendations to make regarding improvement, location, or design.

The vertical lowering-speed variation was never greater than  $\pm 2.5\%$  and generally did not vary by more than  $\pm 1.25\%$  of the average value for

the run. The variation in rotational speed of the output shaft of the variable speed changer was not more than  $\pm 1.5\%$  generally being approximately  $+0.7\%$  of the average value.

One refinement that would have made it faster to determine the setting of the variable speed changer, to give a particular vertical sample lowering speed, would have been a graduated micrometer control for the setting of the variable speed changer.

It should be noted here that different ranges of vertical lowering speeds can be attained by either changing the diameter of the pulley attached to the output shaft of the fixed speed reducer or by exchanging the induction motor for one of a higher or lower speed.

#### Sample Testing Results

The metal single crystals and silver chloride were checked by one or more of the following three methods:

- (1) Chemical etching.
- (2) Cleavage.
- (3) X-ray pictures.

The zinc samples were all tested by the first two methods, and four samples were tested by taking x-ray pictures. Figure 10a shows the angle that the cleavage faces of three different zinc single-crystal samples made with the cylindrical axis of the samples. Figure 10b shows the mirror-like quality of the cleavage faces of zinc single crystals grown in this investigation. The "c" axis is perpendicular to the cleavage plane face. A total of 32 zinc crystallization runs were made with 25 of the runs

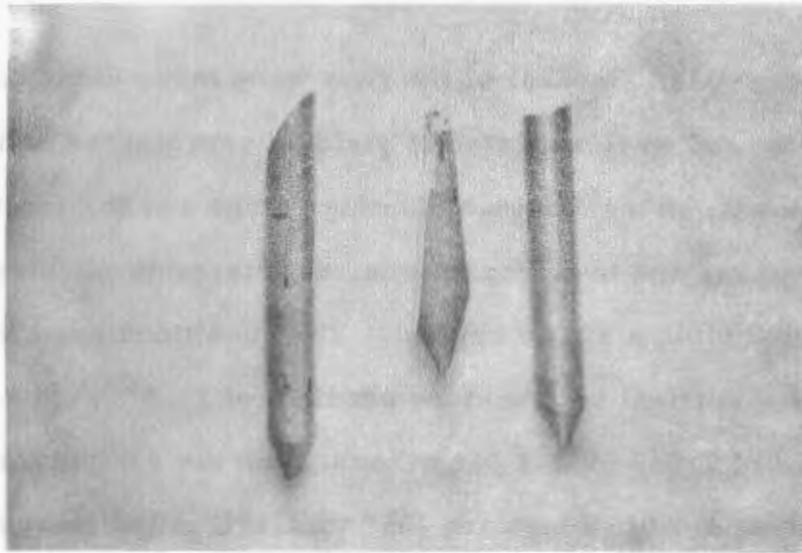


Fig. 10a—Cleavage Faces of Three Different Zinc Single Crystals Showing Varying Angles of Inclination with the Longitudinal Axis of the Sample.

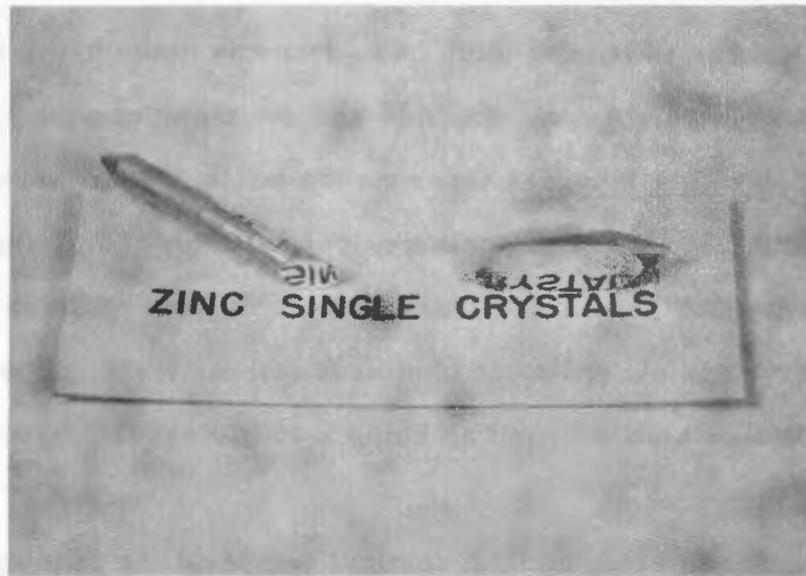


Fig. 10b—Zinc Single Crystal Showing Mirror-Like Cleavage Face.

yielding single crystals. Several of the runs were made using conditions that were extreme and were expected to yield polycrystalline samples. Ten runs were made, using 3/8 inch diameter slugs and the same furnace temperatures and lowering speeds, to determine whether or not each run would yield a single crystal. The conditions used for these runs were a vertical temperature gradient of 11.5°C/cm. and a lowering speed of 0.025 inches per minute, with the sample thermocouple temperature readings between 465° to 471°C at the start of the runs. All ten runs yielded single crystals.

Two lead crystals were grown. The furnace conditions were set to give the same vertical temperature gradient as was used in the zinc runs mentioned above. The lowering speed was adjusted from that used for zinc according to the ratios of densities, heats of crystallization, and thermal conductivities of lead to zinc. One run was made using a 3/4 inch diameter, straight-wall crucible and the other using a 1/2 inch diameter crucible with a 1 degree taper on the walls; the lowering speeds were 0.0102 and 0.036 inches per minute, respectively. Starting temperatures were 368° and 375°C, respectively. Both of these runs yielded single crystals according to chemical etching tests. The large-diameter sample was also verified as being a single crystal by x-ray pictures.

One run was made using tin. A vertical temperature gradient of 17.8°C per centimeter was used for this run. The lowering speed was calculated taking into consideration the density, heat of crystallization, and thermal conductivity of tin compared to zinc as was done in the case

of the lead runs. The diameter of the sample was  $3/4$  inch, the lowering speed was 0.012 inches per minute, and the starting temperature was  $270^{\circ}\text{C}$ . In this case it was first thought that a single crystal was obtained as evidenced by the chemical etch test. However, after removing a small cylindrical sample from the top and re-etching, there was noted to be a very narrow crystal with a low-angle boundary along one side of the sample. This crystal apparently is very thin since no grain is visible when looking at the polished and etched top end of the sample. In any event most of the sample is composed of only one crystal.

Three attempts were made to grow a  $3/8$  inch diameter sample of thallium for Robert Meyerhoff of the Laboratory. In none of the three instances was a single crystal obtained. The temperature gradient varied between 15 and  $19.6^{\circ}\text{C}$  per centimeter, and lowering speeds of 0.0027, 0.0098 and 0.022 inches per minute were used. The lower speeds resulted in multicrystals with grains generally parallel to the longitudinal axis of the sample. These grains appeared to have quite low-angle boundaries with respect to crystallographic orientation. Since thallium undergoes a crystal structure change from a cubic to a hexagonal form in the solid state at  $225^{\circ}\text{C}$ , it was thought that perhaps the adhesion to the crucible walls was causing a stress that resulted in forming new crystals as the transformation took place when the sample passed through this transformation temperature. The samples were all passed through the transformation temperature at the same rate that they were grown.

The data on the silver chloride crystallization runs made for Dr. Richard Reade of the Laboratory are presented in Table II below.

Table II

## Silver Chloride Crystallization Run Data - Graphite Crucibles

Run No.	Sample Diam. (in.)	Vertical Temperature Gradient ( $^{\circ}\text{C}/\text{cm}$ )	Lowering Speed (in./min.)	Type of Crystal
1	3/8	19.7	0.0056	Single
2	9/16	15.5	0.0056	Single
3	9/16	14.4	0.0056	2 crystals
4	Run not completed; sample leaked through crucible walls.			
5*	3/4	16.1	0.0273	Multi-crystal Opaque grains.
6	3/4	21.8	0.0026	Multi-crystal Large opaque grains.

\*--Denotes special run to attempt to produce a polycrystalline sample.

The first two runs indicated a fair degree of success, but the result of Run 6 was disappointing because it was thought that these conditions should yield a single crystal. Dr. Reade thought that perhaps some impurity in the silver chloride may have prevented the formation of a single crystal since there was a brownish layer noted on the top of the sample after the crystallizing run was made. The impurity may have been due to oxidation from the atmosphere or some impurity not removed in the cyanide purification process used to prepare the silver chloride.

In the series of three x-ray pictures taken of each of six different metal samples, which by chemical etching were shown to be single crystals, none gave evidence that the samples were not single crystals.

### EQUIPMENT COSTS

This section on equipment costs was included so that those persons interested in duplicating this unit would have an idea of the approximate costs of the various assemblies in this crystal growing unit. The costs are given in Table III. It is believed that these costs are fairly representative and realistic since commercial units were used in practically all places except for the two furnaces. The furnaces were fabricated by our own shops in order that the desired dimensions and thermocouple accommodations could be obtained. Commercial units of similar construction and temperature ratings would cost approximately \$50.00 more than listed in Table III.

The cost (approximately \$500.00) may seem high, but the current cost of single crystals is approximately \$50.00 to \$100.00. If several crystals were needed, the crystallizer would soon pay for itself.

### CONCLUSIONS

The Bridgman crystallizer, shown and described previously, worked quite satisfactorily in the growing of tin, lead and zinc single crystals.

There are certain modifications which would probably improve the operation or increase the utility of this unit. One change would be to lengthen the furnaces to improve the uniformity of the temperature in

Table III

## Costs of Various Components in the Bridgman Crystallizing Unit

ITEM	COSTS	
<b><u>FURNACE ASSEMBLY</u></b>		
Fabrication and assembly of furnace	\$ 56.34	
Powerstats (2)	29.60	
Power line switches (2)	5.84	
Ammeters (2)	17.00	
Fuses (2)	0.10	
Other components	2.20	
Total-----	<u>\$111.08</u>	<u>\$111.08</u>
<b><u>SAMPLE-LOWERING ASSEMBLY</u></b>		
Metron speed reducer (1936:1)	\$ 75.00	
Metron speed changer (25:1 change)	95.00	
Minneapolis-Honeywell, 30 rpm induction motor	30.00	
Powerstat and capacitor to vary speed of motor	11.40	
On-off and reversing switch for motor	1.84	
Supporting wire and chain	2.00	
Total-----	<u>\$215.24</u>	<u>\$215.24</u>
<b><u>CRYSTALLIZING CRUCIBLE ASSEMBLY</u></b>		
Stainless steel support crucible	\$ 36.50	
Alundum and asbestos insulation	0.50	
Chromel-alumel thermocouple	1.30	
Graphite crucible	2.65	
Stainless steel screws	0.30	
Total-----	<u>\$ 41.25</u>	<u>\$ 41.25</u>
ANGLE IRON FRAME-----	\$ 55.67-----	\$ 55.67
<b><u>ASSEMBLY AND INSTALLATION</u></b>		
COSTS-----	\$ 67.80-----	<u>\$ 67.80</u>
Total Cost of Bridgman Crystallizing Unit-----		<u>\$501.04</u>

the heated zones. This would also permit longer single crystals to be grown. Another change would be the installation of a temperature-controlling mechanism or voltage stabilization unit for both furnaces so that more constant temperatures would be maintained. This would be required if inorganic crystals with their low thermal conductivities and slow lowering-speeds were to be grown into single crystals.

The sample-lowering mechanism gave a quite constant sample-lowering speed that generally did not vary by more than  $\pm 1.5\%$ . The maximum variation was  $\pm 2.5\%$ .

The crystallizer assembly functioned very satisfactorily, and the design method of minimizing horizontal heat transfer and maximizing vertical heat transfer was apparently quite satisfactory in the growing of low melting-point, metal, single crystals. In the case of thallium, it is believed that the reason single crystals were not obtained was due either to the adherence of the sample to portions of the graphite crystallizer or to the fact that there was a change in crystal structure in the solid state, at a temperature  $80^{\circ}\text{C}$  below its melting point, that required different conditions than the growing of the single crystal from its melt.

The unit was found capable of consistently yielding single crystals on duplicate runs using the same operating conditions.