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Research and Development Report

LARGE BRIDGMAN - TYPE UNIT FOR
PREPARING LARGE AND SMALL
DIAMETER SINGLE CRYSTALS

by

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LARGE BRIDGMAN-TYPE UNIT FOR PREPARING LARGE AND SMALL
DIAMETER SINGLE CRYSTALS

Robert E. Slonaker, Jr., Morton Smutz and Edwin H. Olson

ABSTRACT

The design and performance of a large Bridgman-type single crystal growing unit are presented. The unit can be used to grow either single crystals more than 2 in. in diameter, or simultaneously grow several smaller crystals. Dimensioned drawings and photographs are included.

Preliminary tests were made by using zinc of 99.99+% purity. It was demonstrated that single crystals of zinc 2-1/4 in. in diameter by 7-1/4 in. in length could be grown in graphite crucibles having 50° conical nucleating tips. By employing the same operating conditions it was also possible to grow seven single crystals 3/8 in. in diameter by 7-3/8 in. in length, simultaneously.

The unit performed quite satisfactorily, and apparently would be adaptable to growing single crystals of many other low melting materials.

INTRODUCTION

This project is a continuation of work on growing single crystals by the Bridgman method as discussed in a previous report (1). The main interest in building and testing a larger unit was to determine if the conditions found suitable for the growth of single crystals in the small unit would be applicable to a larger unit.

The simultaneous growth of several small diameter single crystals saves time, and produces more uniform single crystals than when the crystals are grown in separate runs.

Large single crystals are in demand for scientific investigation (2, 3, 4). For example, they are required as monochromators in neutron diffraction studies and as analyzing crystals in gamma ray spectrometry.

EQUIPMENT

This larger Bridgman-type single crystal growing unit is similar to the one previously described (1), except that it is larger and the furnace is of modified design. This unit provides for the lowering of a crystallizing crucible assembly at a controlled rate through a temperature gradient maintained within the furnace. A photograph of the complete unit is shown in Fig. 1.

Furnace Assembly

The furnace assembly details, including dimensions, are shown in Fig. 2. An Alundum core is wound with four separate sections of Kanthal A-1 resistance wire. Powdered Dicalite is used for thermal insulation. The furnace assembly is secured by four furnace bolts which hold the transite top and bottom covers against a galvanized cylindrical sheet metal shell.

A. Angle-iron frame (18" x 30" x 51")	D. <u>Sample Lowering Assembly</u>
B. Furnace assembly	1. Motor
C. Powerstats	2. Motor control switches
E. Thermocouples (sample)	3. Variable speed changer
F. Thermocouple wire guide tubes	4. Speed reducer
G. Thermocouple selector switch	5. Sample lowering pulley
	6. Calibrated pulley (sample travel)

Fig. 1 - Photograph of large Bridgman-type crystallizing unit.

The furnace bolts extend from the bottom of the furnace and serve to secure the furnace assembly to the angle-iron supporting frame. The furnace assembly can be leveled by means of taps on these bolts, above and below the frame. Leveling is necessary to ensure that the crystallizing crucible assembly remains in the center of the furnace core while the crucible is lowered. Non-uniform radial cooling within the crucible could result in the formation of a multi-crystal.

The lead wires from the furnace windings are brought out through the bottom transite end piece to facilitate the insertion of the crucible assembly prior to the start of a run.

We found from experience with the small unit (1) that warping of the transite end pieces made it difficult to keep the furnaces level, and the cores in good alignment. For this reason the furnace for the large unit was designed with a single core.

The auxiliary furnace sections were incorporated in order that a more uniform temperature could be maintained in the upper hot zone, and the lower cooler zone of the furnace assembly. The auxiliary furnace sections were wound with 20-gage Kanthal A-1 and the top and bottom furnace sections with 14-gage Kanthal A-1 resistance wire. A 110 volt, single phase, AC electrical supply was used

and the maximum allowable current to each furnace section was estimated to be between 6 and 7 amps. The furnace assembly was designed for a maximum operating temperature of 900° C.

Crystallizing Crucible Assembly

The crystallizing crucible assembly, shown in Fig. 3, consists of a graphite crucible 2-1/4 in. in diameter by 9 in. long, contained in and thermally insulated from a stainless steel crucible holder by 1/16-in. asbestos sheet. A threaded graphite stem protrudes from the bottom of the graphite crucible through the bottom plate of the stainless steel holder. Nucleation and growth of the crystal at the tip are promoted by a graphite heat sink which is screwed to the stem of the graphite crucible. This heat sink promotes the flow of heat downward while the crucible assembly is lowered into the low-temperature region of the furnace.

The thermocouples attached to the stem and to the outer wall of the graphite crucible approximately 8 in. above the base permit a close estimate of the temperature of the zinc sample. The thermocouple wires, electrically insulated with Alundum and/or woven fiberglass insulators, extend through holes in the cap to the lead wire connectors.

An 18 gage chromel A support wire is attached to the cap with a stainless steel, screw-type lock.

Fig. 3. - Crystallizing crucible assembly (large diameter crystals).

The cap and bottom plate are secured to the stainless steel holder with stainless steel screws. Thus the crucible can be changed by merely removing the bottom plate.

Fig. 4 shows both the crystallizing crucible used for growing one large single crystal and that used for growing seven $3/8$ -in. diameter single crystals, simultaneously. Both the small and large diameter crucibles had fifty-degree conical nucleating tips. The small crucibles are tapered, from an inside diameter of $3/8$ in. at the top to $5/16$ in. at the bottom (exclusive of the tip). The larger crucible was tapered from $2-5/16$ in. in diameter at the top to $2-1/4$ in. in diameter at the bottom. The taper facilitates removal of the single crystals.

A Vycor sleeve is used to surround the cluster of seven crystallizing crucibles to reduce the annular space and produce more uniform vertical temperature gradients.

A nonuniform temperature gradient, caused by thermal convection currents, could cause new crystal nuclei to form on the inner crucible wall, resulting in the formation of a multicrystal. In order to determine vertical temperature gradients for the upper and lower sections of the center crucible, a third thermocouple is

Several Small Single
Crystals

One Large Single
Crystal

- A - Graphite crucibles
- B - Graphite heat sink
- C - Asbestos sheet
- D - S. S. bottom plate
- E - Thermocouples
- F - Vycor sleeve

Fig. 4 - Crystallizing crucible assemblies.

centered on the outside wall.

Sample Lowering Assembly

The sample lowering assembly permits the crystalizing crucible assembly to be lowered through the furnace at a constant rate. With reference to Fig. 1, a 20 RPM synchronous motor is connected to a drive pulley through a speed changer and a speed reducer. A ball chain (1/8-in. diameter balls) attached to the drive pulley, passes over an idler and positioning pulley and is connected to the supporting wire of the crucible holder by means of a brass adapter coupling. The speed reducer provides for 1936:1 reduction, and the speed changer permits a 25-fold change in the lowering speed. The sample-lowering motor is wired through a reversing switch, fuse, and an on-off switch mounted on the control panel.

The mount for the sample lowering assembly is bolted to the angle-iron frame with the motor, speed changer and speed reducer in place. A universal joint connects the speed reducer shaft to the drive pulley shaft and prevents large stresses and excessive wear on the speed reducer, should the two shafts be misaligned. By minimizing the stresses due to shaft misalignment, a more uniform sample lowering speed is attained. The

drive pulley is designed so that the crucible holder is lowered 2-1/2 in. per revolution of the pulley. A calibrated scale on a second pulley attached to the drive shaft permits continuous measurement of the vertical distance traveled by the crystallizing crucible assembly.

The maximum and minimum sample lowering speeds attainable were 0.147 and 0.0069 in./min., respectively.

Temperature Measurement

The furnace temperature gradient was determined by measuring the temperatures at the centers (lengthwise) of the two main heating elements. The graphite crucible thermocouples are chromel-alumel double-junction loop-type which permit the determination of average temperatures at the desired points.

A guide tube assembly for thermocouple leads was designed to minimize snagging and to keep the cold junctions away from the hot region above the furnace as the crucible assembly is lowered. The guide tube assembly consists of six - 12 in. small Bakelite guide tubes securely inserted into a Saran block. Pieces of chromel and alumel wire of the same length as the guide tubes are soldered to 26 gage stranded insulated flexible lead wires and are inserted into the guide tubes. The other ends of the chromel and alumel wires are joined to the crucible

thermocouple lead wires by means of small brass couplings. As the crucible assembly is lowered through the furnace, the flexible lead wires are pulled through the guide tubes. At the lowest point of travel, these cold junctions are still inside the guide tubes, approximately 12 in. from the top of the furnace.

All thermocouple leads, including those from the furnace, are connected to terminal blocks mounted on an aluminum plate which is bolted to the top side of the angle-iron frame. The terminal blocks are connected to a thermocouple selector switch on the main control panel. The selector switch is connected to a strip chart recorder for monitoring and recording the temperatures of any of the thermocouples. The cold junction temperature is determined by means of a mercury thermometer attached to one of the guide tubes.

OPERATIONAL PROCEDURE

Sample Preparation

Zinc of 99.99+% purity, as obtained from New Jersey Zinc Company in slab form, was used in all runs. Our original intention was to cast the zinc for the 2-1/4 in. diameter crystals by melting acid-cleaned water-rinsed and dried pieces of zinc directly in the crystallizing crucible using induction heating. After two

trials this procedure was abandoned because of pipe formation in the casting and surface imperfections due to the presence of zinc oxide that failed to migrate to the top of the sample. To circumvent these problems the furnace assembly of the large Bridgman unit was used in the casting operation described below.

A graphite melting crucible with a drain port at the bottom was placed above the crystallizing crucible in the furnace assembly of the large Bridgman unit. A graphite rod plug was used to close the drain port and previously cleaned pieces of zinc were then placed in the melting crucible. Both crucibles were then heated to a temperature approximately 80°C above the melting point of zinc and held at this temperature for approximately 30 minutes to permit most of the zinc oxide to rise to the top surface of the molten zinc. The graphite rod plug was then removed permitting the molten zinc to flow down into the crystallizing crucible. A program of power reductions to the top auxiliary section and to the top and bottom furnace sections caused the zinc to solidify from the cone tip upwards at a relatively slow rate.

This procedure proved to be satisfactory in eliminating surface imperfections and drastically reducing pipe formation in both the large and small diameter

samples. The zinc oxide slag was effectively retained on the inner walls of the melt crucible with virtually no zinc oxide present on the surface of the cast sample in the crystallizing crucible.

The total time required for the casting operation from the start of heating until the zinc was completely solidified was approximately 5-1/2 hrs. Approximate zinc solidification rates were 0.08 and 0.14 inches per minute for the large and small diameter zinc samples, respectively.

Crystallizing Run

The crystallizing crucible assembly shown in Fig. 3 was connected to the ball-type chain of the sample lowering assembly using a brass adapter fitted with set screws. The crucible assembly was first lowered manually to the lowest position in the tube of the furnace that would be reached during the crystallizing run, and then the motor of the sample lowering assembly was used to raise the crystallizing crucible to the starting position for the crystallizing run. This lowering and raising procedure was necessary to eliminate any slack in the ball-type chain that would result in a nonuniform sample lowering speed and to avoid errors in determining the position of the crystallizing crucible assembly during a crystallization run. At the starting

position, the bottom surface of the graphite heat sink was directly opposite the bottom winding of the top furnace section. The calibrated scale on the sample lowering assembly was then set to zero.

Thermocouple wires from the crystallizing crucible assembly were passed through the top transite furnace core cover and then connected to the lead wires in the thermocouple wire guide tube assembly. The connections were made using brass adaptors and set screws to assure good electrical contact. The thermocouple wires were kept as straight as possible to prevent excessive binding or dragging as they were drawn through the holes in the transite furnace cover. Binding or dragging of the thermocouple wires could cause the sample to become off-center in the furnace developing nonuniform vertical temperature gradients in the crystallizing crucible that could result in the formation of new crystal nuclei and subsequent growth of a multi-crystal instead of the desired single crystal.

The zinc was heated rapidly to a temperature 70 to 100° C above its melting point, 420° C, to ensure complete melting of all crystal nuclei. The speed changer was set to give the desired sample lowering speed and the crystallizing run started by turning on

the sample-lowering motor and the electric timer simultaneously. Sample lowering speed was checked periodically during the run by noting the time required for 1/4-in. increments of lowering of the sample. Maximum speed variation noted was $\pm 3.5\%$ of the average, but for most runs it did not exceed $\pm 2.5\%$. The sample was generally lowered 12 inches into the low temperature zone of the furnace and the sample-lowering motor and current to all furnaces turned off. The sample was permitted to cool to 100°C or lower before removing it from the furnace assembly. This permitted slow cooling to minimize thermal stresses which could have an adverse effect on perfection of the single crystal.

Temperatures of the furnaces and the graphite crystallizing crucible were monitored using a strip chart recorder. Graphite crucible temperatures were monitored more closely than the furnace temperatures since the crystallization rate of zinc would be strongly dependent on the vertical temperature gradient in the graphite crystallizing crucible.

Sample Removal and Examination

The crystallizing crucible assembly was removed from the furnace and the stainless steel top cap removed. The removal of the large 2-1/4-in. diameter samples was

effected by simply inverting the assembly slowly and permitting the zinc sample to slide out of the crystallizing crucible under the force of gravity. In the case of the small $3/8$ -in. diameter samples, the individual graphite crystallizing crucibles were removed from the crystallizing crucible assembly by unscrewing the graphite heat sinks from the threaded graphite stem of the crystallizing crucible. The graphite crystallizing crucibles were individually inverted and gently tapped to loosen the zinc sample and permit its easy removal. Since single crystals of zinc are quite ductile and easily bent, they were carefully handled and no large force was used in removing the samples from the crystallizing crucibles.

The same graphite crystallizing crucible was used in all six runs for growing the $2-1/4$ -in. diameter zinc samples. Some of the crucibles used in growing the small $3/8$ -in. diameter samples had to be sacrificed in order to recover the sample. These crucibles were found to have small voids on the inner walls that permitted the zinc sample to become locked in the crucible. In one run a small crystallizing crucible being used for the third time developed voids large enough to permit small beads of zinc to leak through the wall near the bottom

of the crucible. Apparently the original voids became larger due to oxidation and permitted slight leakage of molten zinc to occur during the high temperature preheat portion of the crystallizing run.

The zinc samples were etched using a 1:1 HCl-H₂O (by volume) solution at room temperature for approximately 20 seconds, rinsed with distilled water and examined visually for the presence of grain boundaries. The absence of grain boundaries was taken as evidence that a single crystal had been grown. Several of the single crystals were cleaved and these showed no indication of other internal crystal grains.

EXPERIMENTAL RESULTS

Growth of Large Single Crystals

The heat up procedure established for these runs required approximately 2-1/2 hr. for heating and melting the sample and 2 hr. for reaching essentially steady-state conditions within the apparatus. Temperature conditions at the start of the sample lowering procedure were relatively consistent from run to run in this series, as is shown in Table I.

Fig. 5 shows plots of the temperature at the tip of the graphite crucible vs. the distance traveled by the sample for three different lowering speeds. The

initial change in slope of each of the curves indicates the position where nucleation occurs and the zinc has first started to crystallize. These cooling curve breaks are rather well defined. The second cooling curve breaks that indicate when the last of the zinc has crystallized are not as well defined, especially for the higher speed runs.

It was found, however, that the thermocouple at the top of the crystallizing crucible was highly responsive and could be used to determine accurately when the last of the zinc sample had crystallized. These data were used in determining the average rate of crystallization shown in Table II. Fig. 6 shows how the crystallizing time was determined for a typical run.

Growth of Small Zinc Single Crystals

The simultaneous growth of several small-diameter crystals was studied after the large-diameter single crystal runs had been completed. It was decided to employ the same furnace power settings as were used in the large-diameter single crystal growing runs to determine whether or not different sample lowering speeds would be required. The temperature conditions reached at the start of the sample lowering procedure are shown in Table III.

Table I
Temperature Conditions within Apparatus
at Start of Sample Lowering Procedure
Large-Diameter Zinc Crystals

<u>Temperatures, °C.</u>				
<u>Run No.</u>	<u>Crucible Tip</u>	<u>Crucible Top</u>	<u>Top Furnace</u>	<u>Bottom Furnace</u>
Zn-L-1	491	489	440	315
Zn-L-2	495	495	447	313
Zn-L-3	496	496	440	322
Zn-L-4	494	494	446	319
Zn-L-5	499	499	447	316
Zn-L-6	489	498	446	317

Fig. 5 - Plot of temperature at the tip of the graphite crystallizing crucible vs. sample lowering distance for 2-1/4-in. diameter, zinc, crystal growing runs.

TABLE II

Summary of Data for Growing Large Diameter Zinc Crystals

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Vertical Temperature Gradients

Run No.	Furnace		Crystallizing Crucible		Sample Lowering Speed (in./min.)	Crystal-lization Rate*** (in./min.)	Type of Crystal (single or multi)
	Nucleation (°C/in.)	Final Crystal (°C/in.)	Nucleation (°C/in.)	Final Crystal (°C/in.)			
Zn-L-1	12.2	9.5	3.6	3.8	0.0069	0.0180	Single
Zn-L-6	12.4	9.8	*	*	0.0107	0.0221	Single
Zn-L-5	12.7	10.5	4.4	4.6	0.0110	0.0265	Single
Zn-L-4	12.5	10.2	4.2	4.4	0.0151	0.0292	Multi
Zn-L-3	9.2	8.1	4.7	4.9	0.0338	0.0435	Multi
Zn-L-2	**	**	**	**	0.147	**	Multi

*No values reported because of faulty thermocouple readings.

**Unable to report. Sample did not crystallize during the time it was lowered the 12 in. maximum permitted in the crystallizing unit.

***Based on a length of zinc assuming a 2-9/32-in. diameter cylindrical sample.

Fig. 6 - Plot of temperatures at tip and top of graphite crystallizing crucible vs. sample lowering distance for 2-1/4-in. diameter, zinc, single crystal growing run Zn-L-5.

A total of three runs was made as shown in Table IV. The first two runs were made using a sample lowering speed of 0.011 in./min. which, in the case of the large-diameter samples, was found to result in single crystals. The third run was made at a speed of 0.015 in./min., since, at this speed, a multi-crystal of zinc was found to be formed in runs with the large samples. The first run was successful in producing seven individual single crystal samples, $3/8$ in. in diameter at the top, $5/16$ in. at the bottom, and $7-3/8$ in. in total length. The second and third runs also resulted in single crystals of the same diameter measurements but $6-1/8$ in. in total length. The shorter length of these samples was due to a change in casting crucible arrangement which was found necessary to prevent bridging of the zinc samples at the top after the casting operation.

The average crystallization rate was determined by noting the time of breaks in the cooling curves as described in the previous section with crystallizing times determined as illustrated in Fig. 7. The breaks were less well defined, however, because less zinc underwent solidification.

Table III

Temperature Conditions within Apparatus
at Start of Sample Lowering Procedure

Small-Diameter Zinc Crystals

Temperatures, °C.

<u>Run No.</u>	<u>Crucible Tip</u>	<u>1/2 Vertical Distance along Crucible</u>	<u>Crucible Top</u>	<u>Top Furnace</u>	<u>Bottom Furnace</u>
Zn-Mu-1	481	487	481	427	332
Zn-Mu-2	481	478	478	426	327
Zn-Mu-3	486	494	495	446	322

Table IV

Summary of Data for Growing Seven Zinc Crystals Simultaneously
Vertical Temperature Gradients

Run No.	Furnace		Crystallizing Crucible		Sample Lowering Speed (in./min.)	Crystal-lization Rate* (in./min.)	Type of Crystal (Single or Multi)
	Nucleation (°C/in.)	Final Crystal (°C/in.)	Nucleation (°C/in.)	Final Crystal (°C/in.)			
Zn-Mu-1	14.9	11.7	2.98	3.50	0.0109	0.0267	All Single
Zn-Mu-2	14.2	12.3	2.95	3.25	0.0113	0.0327	All Single
Zn-Mu-3	14.0	11.7	3.45	4.10	0.0150	0.0330	All Single

*Due to difficulty in determining accurately when the samples nucleated these values should be regarded only as approximate average crystallizing rates.

Fig. 7 - Plot of temperatures at the tip, middle and top of the graphite crystallizing crucible vs. the sample lowering distance for the 3/8-in. diameter, zinc, single crystal growing run - Zn-Mu-2.

CONCLUSIONS

1. This unit is capable of growing large single crystals of zinc, 2-1/4 in. in diameter by approximately 7-1/4 in. in length, or seven smaller single crystals simultaneously, each 3/8 in. in diameter and from 6-1/8 to 7-3/8 in. in length.
2. Using the maximum vertical temperature gradient attainable in the furnace assembly of approximately 14°C./in. and lowering speeds up to 0.011 in./min., single crystals of zinc 2-1/4 in. in diameter by 7-1/4 in. in length were obtained. At a lowering speed of 0.011 in./min. the actual crystallizing rate was found to be approximately 0.0280 in./min. When the lowering speed was increased to 0.015 in./min. or faster, multi-crystal zinc samples were obtained. Using the same furnace conditions as for the large-diameter zinc samples, it was found that seven small 3/8 in. diameter single crystals of zinc could be grown simultaneously at speeds of 0.011 and 0.015 in./min., and the corresponding crystallizing rates were approximately 0.030 and 0.033 in./min. respectively.

An upper limit of the lowering speed that could be used to grow seven small-diameter single crystals simultaneously was not determined.

3. The furnace assembly was found to have a high thermal capacity and to show a considerable temperature lag on rapid heat-up. This necessitated programming the power to the furnaces to avoid reaching higher temperatures than desired when starting a single crystal growing run. The high thermal capacity, however, was desirable, since it imparted a high degree of thermal stability which is quite advantageous when growing single crystals from their melts.
4. The sample lowering assembly provided the uniform sample lowering speeds needed.
5. The crystallizing crucible assembly was apparently satisfactory, since no difficulty was experienced in obtaining single crystals using replicated conditions.
6. The temperature measurement technique was satisfactory for determining the cooling curve breaks and average crystallizing rates for the large-diameter zinc crystal growing runs. In the case of several small-diameter zinc crystals grown simultaneously,

however, a more sensitive temperature measurement method is required so that the sample nucleation point can be more accurately determined. A "differential" or "series" thermocouple arrangement might be successful in amplifying the temperature changes at nucleation and result in a better-defined cooling curve break.

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