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Techniques for Growing and Mounting Small Single Crystals of Refractory Compounds

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An apparatus is described for growing single crystals in a small droplet of melt at temperatures up to 1,600° C. The droplet, held by capillarity at the junction of a thermocouple, can be observed during the process through a binocular microscope. The heating is by means of a high-frequency current that can be separated by a filter from the thermocouple electromotive force, allowing the electromotive force to be measured continuously. The apparatus is therefore suitable also for determining melting points.

The process of mounting and grinding the single-crystal specimens for X-ray diffraction and a simple but versatile micromanipulator to facilitate these procedures are described.

1. Introduction

A necessary preliminary to single-crystal X-ray diffraction studies is the procurement of a suitable single-crystal specimen. The ideal specimen should be pure, free from flaws or twinning, and well enough developed that the crystallographic axes can be found from the orientation of edges and faces. In spite of of the strictness of these requirements, usable crystals of many substances can be found in natural minerals, or in products of manufacture, because large size is not necessary. Even a crystal that is only 0.05 to 0.1 mm in least dimension can be used with ordinary X-ray cameras. Furthermore, if the external development is poor, the crystal can be oriented by X-ray patterns.

It sometimes is found, however, that all available specimens of a mineral are either too impure or too finely crystallized and that synthetic melts of the proper composition, heated in a furnace, likewise form crystals that are too small. This is the situation among the calcium silicates amd aluminates of portland cement clinker, which are being studied at the Bureau.

Previous experience in preparing X-ray specimens, from melts at low temperatures [1, 13, 18],[2] indicated that the most convenient method is manually controlled cooling of the smallest droplet that will yield a crystal large enough to be used. The small size of the droplet minimizes the number of nuclei and the accidental temperature inequalities that lead to growth of unwanted crystals. More important, the droplets can be observed by means of a stereoscopic microscope during the entire process of crystallization. Twins or other extraneous crystals can be detected as soon as they form, and melted away so that growth of the desired crystal can be recommenced immediately. A much larger body of melt, which would be necessary if the crystals were to be grown for optical or piezoelectric use, is actually an inconvenience when they need be only large enough for X-ray diffraction. To take advantage of this fact, a special apparatus was assembled for recrystallizing small samples at high temperatures under the stereoscopic microscope.

In order that the subsequent operations of mounting the specimen and grinding off excessive glass or crystalline material might also be carried out conveniently under the microscope, a micromanipulator and certain accessories were constructed. The methods described are not claimed to be unique solutions of the problem, but they are felt to be improved in certain details over the techniques seen elsewhere.

2. Apparatus for Crystal Growing

2.1 Hot-Wire Apparatus

Numerous publications have described small furnaces in which a sample can be placed [7, 15, 28, 30] and electric heaters on which the sample can be mounted directly [6, 12, 17, 21] for microscopic observation. In the latter form of apparatus, elaborate insulation is not necessary to protect the microscope objectives if the hot zone is made small enough. This fact makes possible simpler construction and less restriction on the angle from which the sample can be viewed. An apparatus of the directly heated type was therefore constructed. The heater consisted simply of a 5-cm length of 0.3-mm platinum wire bent to a sharp V and supported at the ends by suitable binding posts. The wire was heated by current from a 6.3-v transformer, the primary voltage of which was controlled by a continuously variable autotransformer or a foot-operated rheostat intended for use with sewing machines. The droplet of melt, held by capillarity at the point of the V, was observed through a stereoscopic microscope. A variable-density filter, originally intended for the eyepieces of a pair of binoculars, served to protect the observer from excessive glare.

This hot-wire apparatus, when protected from drafts, was convenient for controlled crystallization at temperatures up to 1,500° C. It provided no indication of the temperature, however, except the reading on an ammeter or voltmeter in the circuit and the behavior of the sample itself. The autotransformer settings or meter readings corresponding

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[2] Figures in brackets indicate the literature references at the end of this paper.

to the melting range could be found by experiment, but when the temperature was high these settings gradually changed as the platinum wire volatilized. Furthermore, it was impossible without an independent measure of temperature to detect small fluctuations due to air currents or changes in line voltage.

2.2. Hot-Thermocouple Apparatus

a. Construction

In considering the addition of a device for measuring the sample temperature, a thermocouple was preferred over a resistance thermometer or radiation pyrometer because reasonable accuracy could be obtained without special calibration of the sensitive element, and suitable measuring instruments were already available.

Attempts to measure the sample temperature with a thermocouple were discouraging because it conducted heat from the sample. Even when made from wire only 0.08 mm in diameter it greatly lowered the temperature. The possibility of supplying heat to the thermocouple from a separate source in order to counteract conduction from the sample was considered. The more suitable procedure finally discovered, however, was to use high-frequency alternating current for the heating, so that the direct-current electromotive force produced by the thermocouple could be separated out by a filter and measured independently. If this is done, obviously the hot-wire arrangement need not be a four-terminal device. The thermocouple itself can also serve as a heating element and support for the sample.

The essential parts that were assembled for the hot-thermocouple apparatus are indicated in figure 1. The heater current is obtained from a 50-w audio amplifier of the type used in public address systems. The amplifier is fed by a Hewlett-Packard model

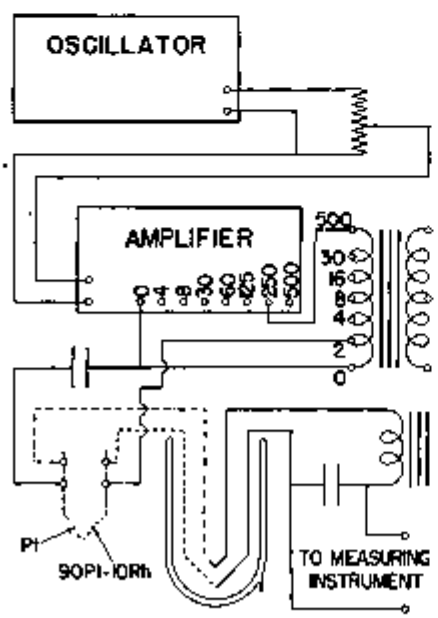

FIGURE 1. *Circuit diagram for hot-thermocouple apparatus.*

200BR audio oscillator through a 10-turn helical potentiometer, which is used as a voltage divider for fine control. This equipment, having been chosen primarily because it was readily available, is much more elaborate than necessary. As the output need be only 25 to 30 w and freedom from harmonics is not important, a power supply built specially for the purpose could be simple and inexpensive.

The power for heating the thermocouple is fed from the output terminals of the amplifier through an additional 50-w audio output transformer, whose secondary is used alone as an autotransformer. To prevent the transformer from acting as a short circuit for the thermocouple electromotive force, a blocking condenser must be inserted in series with the winding. The blocking condenser in use consists of three standard 10-μf oil-filled units connected in parallel.

The blocking condenser adds a considerable reactive component to the impedance of the amplifier load. For most efficient power transfer it is desirable to balance the capacitance by means of an equal inductive reactance in series with the load or reflected through the output transformer; that is, the load should be a series resonant circuit at the frequency used. In addition, the output transformer should of course have the proper turns ratio for the usual match of resistive impedances. These adjustments have not been accurately made in the present apparatus because it is found that sufficient heating can be obtained by the amplifier and transformer impedances shown in figure 1, with an oscillator frequency of about 4.7 kc. The resistance of the thermocouple is 0.1 to 0.4 ohm, depending on its temperature, and the reactance of the condenser is 1.1 ohms at the operating frequency. More careful matching to these values would be necessary if an amplifier of lower power were used. If a low-frequency power supply such as a 400-cycle rotary converter or a 60-cycle transformer were to be used, the impedance match would be extremely important because of the high reactance of the condenser at low frequencies. The use of 60-cycle power would probably be rendered impractical by the large size of the components required.

The thermocouple itself is mounted on an interchangeable four-prong plug-in assembly. The four-prong plug is made by cementing together two standard double banana plugs which fit into a pair of double banana jacks fastened permanently in position under the stereoscopic microscope, as shown in figure 2. In order to replace the thermocouple with a clean one for preparing a new melt, it is necessary merely to plug in a new assembly.

The portion of the thermocouple that is actually heated, at the junction, consists of 2-cm lengths of 0.25-mm platinum and platinum–10-percent rhodium wire. To reduce heating of the connectors these short lengths of fine wire are welded to 0.6-mm extension leads of the same metals, which in turn make contact with the banana plugs. Several of the interchangeable assemblies can be made up in advance by welding new lengths of fine thermo-

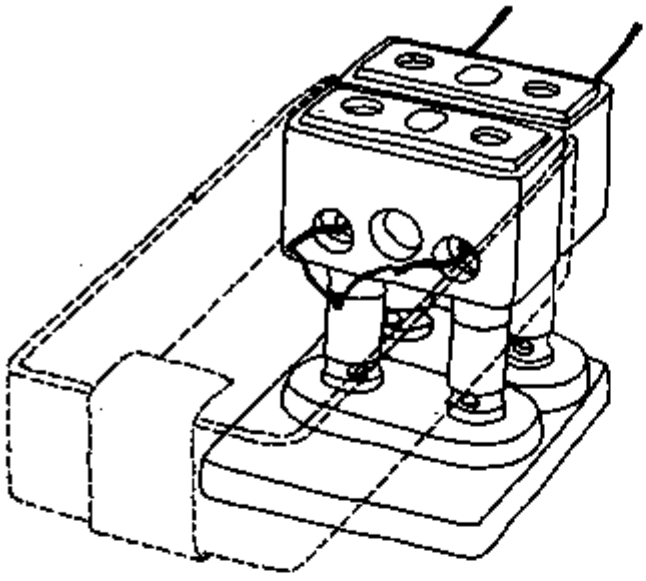

FIGURE 2. *Thermocouple assembly.*

couple wire onto the heavier leads. The leads need not be replaced, as they are not contaminated by contact with the melt; the amount of platinum used per sample is therefore only about 0.06 g.

The thermocouple is protected from drafts by two sheets of mica supported by a brass frame, which clips onto the Bakelite body of one of the double banana plugs, as shown by dotted lines in figure 2. Without the mica cover no special protection for the microscope is necessary if the vertical convection current from the hot wire is avoided, but with it even this danger to the objectives is eliminated.

The heater current reaches the thermocouple through the nearer pair of banana jacks shown in figure 2. The jacks farther from the thermocouple, which are less affected by the heat, can be used for the measuring circuit if convenient interchange of thermocouple assemblies is desired. For the most careful work two extension leads made of the thermocouple metals are connected directly to the thermocouple leads by means of the clamping screws in the second pair of banana plugs. This arrangement, which eliminates contact potentials between the leads and the banana plugs, is the one indicated in the figure.

The measuring circuit is conventional except for the inclusion of the low-pass filter (fig. 1) to protect the measuring instrument from the a-c potential across the thermocouple, which may be as high as 3 v. The filter must not be interposed between the thermocouple and the cold junction, of course, if the extension leads are to serve their purpose. In order that a millivoltmeter pyrometer that requires a low d-c resistance may be used for temperature measurements, the primary of a 100-w radio power transformer, having an inductance of 0.6 h and a resistance of 13 ohms, is employed as the filter choke. The filter condenser consists of two 2-μf oil-filled units in parallel.

All condensers in the heating and measuring circuits must be of the nonelectrolytic type with essentially zero conductance in either direction. The slight rectifying action of an electrolytic condenser could lead to a comparatively large error in the measurement of temperature.

b. Calibration

The convenience of the hot-thermocouple apparatus for its original purpose of growing small single crystals was immediately obvious when it was put into operation. In one of the first experiments a lime-alumina melt in the primary phase region for tricalcium aluminate produced a crystal of that compound better in size and perfection than had been found possible with any other method. The main value of the temperature measurement in growing crystals is for precise control during one experiment lasting up to 2 hr. Reproducibility of readings with duplicate samples on the same thermocouple or with duplicate thermocouples is less important. In view of the other possible uses for the apparatus, however, the absolute accuracy of the temperature measurements was tested as well as the precision of control.

For these tests the oscillator and amplifier were powered by a Sola constant-voltage transformer. A potentiometer whose smallest scale division is 0.001 mv and a Leeds & Northrup Speedomax recorder whose smallest scale division is 0.05 mv were connected so that either might be used at will. The recorder, which prints one point in 3 sec, follows more rapid fluctuations than the galvanometer used with the potentiometer. When the apparatus was carefully protected from drafts, with the thermocouple at about 1,000° C, the minimum fluctuation in the recorded electromotive force over 10-min periods was ± 0.02 mv. As the average potentiometer readings agreed with those of the recorder to within less than 0.02 mv at this and several lower temperatures, the thermocouple electromotive forces were read thereafter from the recorder chart.

Three of the interchangeable thermocouple assemblies were made from single lengths of wire 0.25 mm in diameter. These lengths were taken from 100-foot rolls that had been purchased by the Chemistry Division of the Bureau under strict specifications for homogeneity. Calibration certificates were available for couples made from each end of the original lengths of wire; their agreement within the limits of error of the calibration indicated the corrections to be valid for all couples made from this wire. The corrections amounted at most to +8.5 deg, at an electromotive force of 15 mv, over the temperatures read from a standard reference table of thermocouple potentials [23].

The three thermocouples were employed, with a correction chart, to determine the melting points of five inorganic compounds. The first two couples were used with barium disilicate and the third with potassium sulfate, sodium sulfate, potassium chloride, and sodium chloride, in that order. The barium

disilicate was a sample synthesized as a melting-point standard by Eubank [8], and the other salts were of reagent grade. It was found most convenient in preparing for each determination to grind the substance in an agate mortar, moisten the powder slightly with reagent grade xylene, and transfer a small amount of the paste to the thermocouple junction with a microspatula. All the substances that have been tested adhere satisfactorily when treated in this way except potassium sulfate, which decrepitates at the β-α transition point.

The melting points were determined with the apparatus in a closed room but without the use of any shield against drafts except the two sheets of mica in their holder. Under these conditions the recorder showed random fluctuations up to ± 0.05 mv, or about 4 deg at 1,420° C. The results of the experiments, listed in table 1, indicate a maximum error of ± 5 deg C.

TABLE 1. *Melting-point determinations with the hot-thermocouple apparatus*

Compound	Melting point found	Melting point given in the literature [24]
	°C	°C
KCl	768	770
NaCl	795	800.4
Na_2SO_4	887	884
K_2SO_4	1,064	1,069.1
$BaO.2SiO_2$	1,423	1,418

The value listed for barium disilicate is an average of three determinations—two made with different thermocouples on samples with no preliminary heat treatment, and the third after holding the sample for 4 hr at 1,420° C. All three estimates agreed within 0.02 mv, or less than 2 deg.

c. Other Applications

The performance of the apparatus in these experiments indicated it to be satisfactory in the present form for determining melting points over the working range of the platinum and platinum-rhodium thermocouple wire, with an accuracy of ± 5 deg even at 1,420° C. If greater precision is desired, the high-frequency power supply can be equipped with automatic regulation to decrease the amplitude of the random fluctuations in temperature. Any of the well-known methods of regulating heater voltage or current, or the resistance of the heating element [20], is obviously applicable; perhaps the most desirable regulator would be one sensitive to the thermocouple electromotive force, using the methods of converting the direct to alternating current, and of amplifying minute a-c voltages, which are well established in industrial controller designs.

In considering the use of the hot-thermocouple apparatus for precise determination of a melting point or other transition temperature, several factors must be taken into account. Volatility of the substance renders the method useless if it causes the sample to disappear or to change significantly in composition during an experiment. Sodium chloride, for instance, vaporizes rapidly enough to necessitate some haste in the melting-point determination. Corrosion of the thermocouple by the molten sample also renders the method useless if the composition of the sample changes significantly. The effect of slight contamination on the electromotive force of the thermocouple is probably less important as there is little temperature gradient within the contaminated portion of the wire near the junction.

The thermocouple electromotive force is an accurate measure of the temperature at the center of the junction, but a comparatively large temperature gradient exists between the surface of the wire and the outer surface of the sample. This gradient is actually a help in growing single crystals, and causes no error in the determination of true melting points if the behavior of the material nearest the junction is observed. It may lead to segregation, however, if the solid and liquid have different compositions. For this reason the apparatus probably should be used in the investigation of multicomponent phase diagrams only for a rapid survey preliminary to detailed investigations by other methods. Bending the wire in such a way as to form a radiation shield [19] might well eliminate the temperature gradient within the sample, although probably at the cost of visibility.

A temperature gradient is to be expected from the center of the thermocouple wire to its surface. The maximum temperature difference within the wire may be calculated approximately by the formula [25]

$$\Delta T = qr^2/4k,$$

where q is the rate of heat generation per unit volume of the wire, r is the radius of the wire, and k is its heat conductivity. The least favorable values of these quantities in the present apparatus may be taken as $q = 3.9 \times 10^3$ cal/sec cm^3 (corresponding to the 8 w dissipated per centimeter of wire at 1,450° C), $r = 0.0125$ cm, and $k = 0.07$ cal/sec cm deg (reported by Barratt [3] for platinum–10-percent rhodium at room temperature). The temperature difference calculated from these values is 2.2 deg C. As this is less than the experimental error, the temperature gradient within the wire has been ignored in practice. The skin effect of alternating current tends to reduce the temperature gradient, but that effect is negligible at the frequency used in the present apparatus.

The principle used in this apparatus, heating a thermocouple by an alternating current that is filtered out so that the thermocouple electromotive force can be measured alone, has been described in the literature [5, 14] as long ago as 1919. The idea still seems to offer possibilities in untried applications, such as the measurement of surface temperatures [22], optical pyrometry [9], and elimination of errors caused by the heat conductivity of thermocouples [29].

3. Mounting Specimens

When a single crystal has been grown in a droplet of melt, the preparation is cooled to room temperature as rapidly as possible in order to minimize fur-

ther crystallization. The bead of glass is readily broken off the wire by bending the latter away from the bead with fine pliers. Then it is necessary to mount the specimen in proper orientation on the usual glass fiber attached to a brass pin that fits the goniometer head [32] of the X-ray apparatus.

Orienting of the crystal is facilitated by the visibility of faces and edges during crystallization. These often become more difficult to see after the specimen cools, but, if necessary, the interfering effect of refraction at the surface of the glass surrounding the crystal can be overcome by immersion in a suitable index liquid.

Before the crystal is mounted, the glass fiber is attached to the goniometer pin. The adhesive best suited for this purpose and for mounting crystals stable at fairly high temperatures is jewelers' shellac, which wets the surfaces readily when melted and hardens immediately on cooling. The fiber is cemented in place with the aid of a hand-held hot wire [26, p. 198].

It is desirable that the glass fiber be capable of some tilting relative to the goniometer pin in case the accidental misorientation of the crystal is not fully correctible by means of the movable arcs on the goniometer head. Frequently this has been done by softening the cement holding the fiber on the pin; another method has been the use of a goniometer pin with a thin, flexible section near the end [18, 27]. The method used is to solder on the goniometer pin a 15-mm length of 0.4-mm (0.016-in.) copper wire, as shown in figure 3. The wire is amply rigid to support the fiber and specimen, yet it can easily be bent with fine pliers or tweezers when necessary. The advantage of this arrangement is that tilting of the crystal can be done by displacing the lower end of the fiber, which is attached to the wire. Thus the crystal is not decentered while being tilted, as it is when the goniometer pin itself bends.

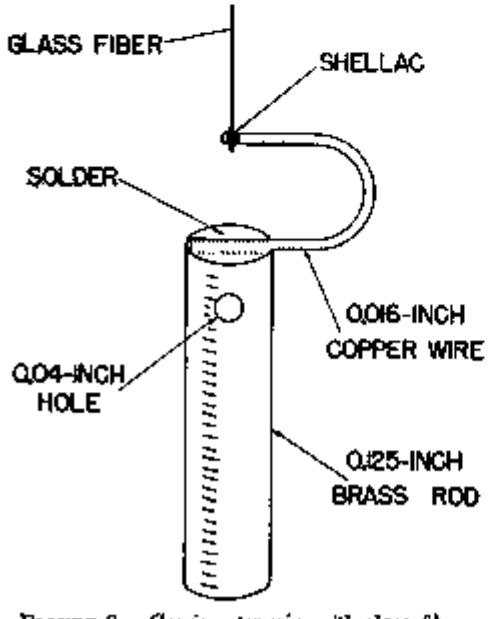

FIGURE 3. *Goniometer pin with glass fiber.*

When the fiber has been attached to the goniometer pin, a small amount of shellac is melted onto the upper end. The next step is then to hold this end of the fiber against the specimen in the proper orientation, heat until the shellac adheres, and allow to cool in the same position. The heating is done with a tiny hot-wire tool made from a 5-mm length of 0.015-mm platinum wire, which is connected to the 6.3-v transformer in series with a 22-ohm resistor.

4. Micromanipulator

To facilitate the procedure of mounting the specimen, some form of micromanipulator is desirable. Most of those described in the literature are either designed for use with a mono-objective microscope at high magnifications [2; 4; 10; 16, p. 62–72], and therefore delicate and limited in range, or adapted to coarser work but difficult to construct [11; 26, p. 202–4]. An attempt was therefore made to design an apparatus that would serve the purpose and yet require as little precise machining as possible in construction. The apparatus was intended for use with a stereoscopic microscope on a vertical-pillar stand with horizontal extension arm and weighted base. It will be described in detail in the hope that the description may be useful to others and stimulate improvements in the kinematic design [26, 31] of a simple manipulator.

The complete apparatus is shown in figure 4. It consists of a base plate, A; two manipulators, B; and a three-legged platform, C. The base plate and the other flat pieces are cut from a ½-inch steel plate, so that their weight insures stability.

Each of the manipulators, B, is in principle simply an ordinary laboratory tripod stand whose base is a 45° right triangle with leveling screws at the vertices. The ends of these adjusting screws are positioned by six small blocks bearing 90° V-grooves, mounted on the base plate. In each set of three blocks, two are turned so that their grooves are parallel, and the third has its groove perpendicular to the direction of the other two. Each manipulator is thereby constrained to a single definite position at all times.

Platform C is an equilateral triangle. When set in place on the base plate its legs are positioned by the three larger V-blocks so that the center of the triangle is almost directly above one of the adjusting screws of each manipulator. Objects can be placed on the platform and operated upon with microtools attached to the jointed column that is mounted on each of the manipulators.

The tools are usually moved by means of the adjusting screws at the sides and at the rear. Each screw causes its manipulator base to tilt about a line between the other two screws. The geometry of the arrangement is such that the tilts produced by the screws at the side and at the rear correspond to horizontal motions of the microtools suspended above the center of the triangular platform. As the screw below the center of the platform is at the 90° vertex of each manipulator base, the horizontal motions are at right angles. The screw at the rear moves

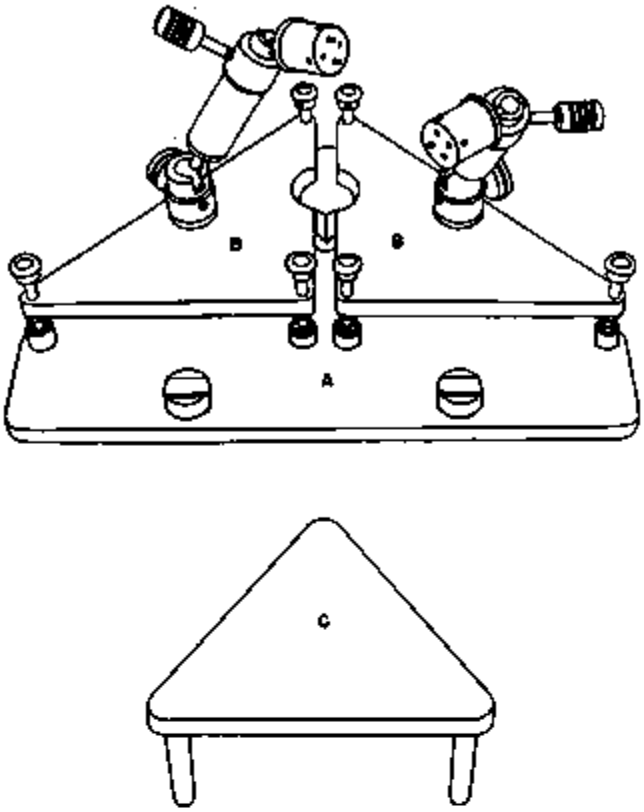

FIGURE 4. *Micromanipulator.*
A, Base plate; B, manipulator; C, platform.

the tool back and forth, and the one at the side moves it from side to side.

As the height of the tool above the base plate is approximately equal to each leg of the 45° right triangle, the motion of the tool is equal in sensitivity and maximum travel to the motion of the corresponding corner of the manipulator base along the adjusting screw. The present model has a sensitivity of 0.08 cm per turn and a maximum travel of about 2.5 cm.

Although the two horizontal motions are independent, a vertical movement requires simultaneous turning of all three adjusting screws. Therefore, it is ordinarily most convenient to adjust the height beforehand and use only the horizontal motions if possible.

The column for supporting the tools, which is mounted on each manipulator, has two ball-and-socket joints (tripod tilt tops, which may be obtained from most photographic supply houses). The two joints are connected by a length of 1-in. round aluminum rod, suitably threaded. At the upper end is a shorter length of the aluminum rod, drilled and equipped with setscrews to hold the ⅛-in. goniometer pins. All the tools used with the micromanipulator are mounted on ¼-in. brass rods so that they can be attached in the same way.

The use of the ball-and-socket joints has the great advantage that tightening them does not displace the object being adjusted, and the chance of accidental damage to delicate specimens is minimized. As they are loosened during adjustment, there is no tendency to spring back out of position as with friction joints. It is desirable that all four ball joints be capable of turning to any direction within a complete hemisphere. Two of those in the present model are not, and the resulting limitation in flexibility is occasionally inconvenient, even though they are used in the lower positions.

The adjusting screws, which are ¼ in. in diameter and have 32 threads per inch, are made with a conical recess in the lower end. This 90° cone rests on a steel ball ¼ in. in diameter, which is positioned by the V-block. The combination provides a kinematic design in which the smooth, hard surface of the steel ball minimizes chattering when the screws are turned. Furthermore, if this surface becomes worn or damaged the steel ball can be replaced very cheaply. A short piece of flexible plastic tubing is slipped over each V-block to prevent the steel balls from rolling off when one of the manipulators is lifted from the base plate.

The adjusting screws were cut on a lathe to insure that the screw threads would be coaxial with the conical surface; this was the only precise machining used in constructing the assembly. The corresponding threads in the manipulator bases were made by hand with a standard tap.

The screw arrangement has disadvantages, being kinematically over-specified and therefore tending to allow lateral movement unless made to a very fine tolerance. This tendency was overcome to a considerable extent by preloading each screw with the simple device shown in figure 5, consisting of a leaf of $\frac{1}{16}$-in. phosphor bronze with two tapped holes and a pointed setscrew. The tool can still be moved laterally by almost 0.001 in. if a horizontal force is deliberately exerted on one of the adjusting knobs, but the force required is great enough so that in practice no accidental motion occurs. Pos-

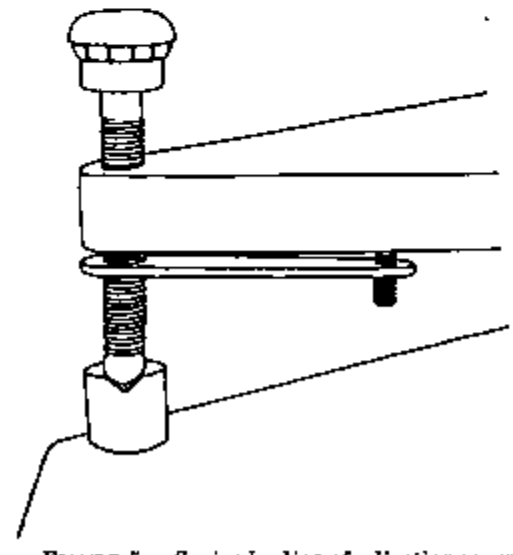

FIGURE 5. *Spring loading of adjusting screw.*

sibly other methods can be devised to eliminate the lateral movement entirely.

The manipulator has been used for almost a year as a permanent adjunct to the stereoscopic microscope. It has proved versatile and convenient for magnifications from ×5.4 to ×90. On one occasion, a standard petrographic microscope was set on the triangular table, and the manipulator was used to operate an implement on its stage at about ×150. Even under these conditions the precision and freedom from vibration were satisfactory, although the increased height made the arrangement cumbersome.

The present model is less sensitive to vibration than the manipulator described by Strong [26, p. 202–4], probably because of the rigidity of the 1-in. aluminum rod used for the jointed column. Furthermore, the strength of the ball-and-socket joint when clamped makes it capable of supporting much heavier objects; even a small electric-motor grinder weighing more than 200 g has been used as an implement.

It is believed that the simplicity, ruggedness, and cheapness of this apparatus make it valuable in many uses for which more elaborate equipment is not absolutely necessary, in addition to the mounting of crystals.

5. Grinding

The specimen containing a desired crystal may also contain extra crystals or excessive amounts of glass. If so, it is necessary to remove the undesirable portions of the specimen and leave the crystal undamaged. This can best be done by mounting the specimen as usual, in such a position that the undesirable portion is exposed, and then carefully removing that portion. A similar procedure of mounting and then removing the excess is necessary if cylindrical specimens are desired to simplify absorption corrections.

The slow removal of a part of the specimen is often done by hand with a solvent applied on a camel's-hair brush. In some cases no suitable solvent can be found, and mechanical removal becomes necessary. A small grinder to be used for this purpose on the micromanipulator was made from a miniature 27.5-v Alnico field motor, by fitting a simple adapter on the shaft so that standard abrasive tips with ⅛-in. shanks could be used. The motor operates satisfactorily on 120-v 60-cycle alternating current, with a 25-watt bulb and selenium rectifier in series and a 1,000-microfarad electrolytic condenser in parallel. The best grinding surface was obtained by stirring a pinch of 5-μ diamond powder into a few drops of thinned lacquer and coating a ⅛-in. polished steel shaft with the mixture. A surface thus prepared was found to cut many times faster than any other that has been tried. In addition, its relative smoothness caused much less vibration of the specimen than had previously been considered unavoidable, reducing considerably the chance of accidental breakage or loss. The use of diamond powder in other types of crystal-grinding apparatus, such as that of Sturdivant [27] for automatically producing a cylinder, would probably have similar advantages; its added convenience makes the effort of procurement worthwhile, and its cost per specimen is slight.

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